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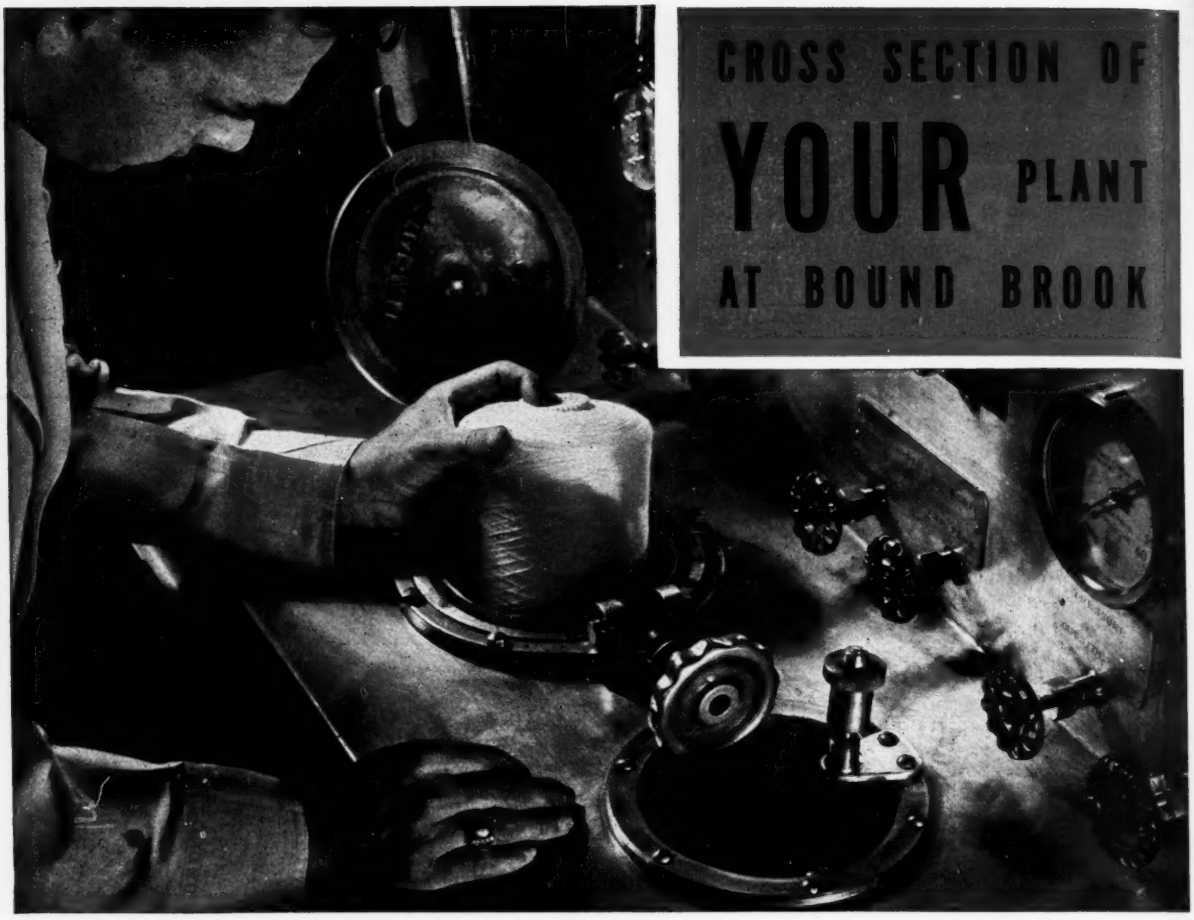

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PHOTOCHEMICAL REACTIONS IN SILK

HENRY A. RUTHERFORD and MILTON HARRIS

ABSTRACT

Silk is more readily deteriorated by the action of light than any other natural textile fiber. While the nature of the deterioration has not been clear, it has been known that the rate of decomposition may be considerably altered by pretreatment of the fiber with solutions of various chemical compounds. For example, silk treated with dilute solutions of basic compounds is more stable toward the action of light than untreated silk, whereas silk treated with dilute solutions of mineral acids exhibits greatly decreased stability under the same conditions.

An investigation by Research Associates of the Textile Foundation at the National Bureau of Standards of the influence of a number of reagents on the photochemical deterioration of silk now reveals that the rate of deterioration is in part influenced by the cationic content of the silk. The removal of cationic substances, by two independent methods results in practically identical decreases in the stability of the fibers to light. On the other hand, the stability is readily restored by treatment with basic compounds. Inorganic bases produce a number of undesirable effects, such as a harsh "feel" and a tendency for the cloth to yellow. These effects can be eliminated by the use of any one of a number of different organic basic compounds.

It was further shown that the inactivation of the carboxylic acid groups of silk by methylation with diazomethane does not appreciably change the photochemical behavior of the fiber but that the methylation of only a small percentage of the hydroxyl groups of tyrosine results in a marked increase in photochemical stability. The results suggest that alteration of the tyrosine hydroxyl groups by reaction with various reagents is responsible for at least part of these stabilizing effects.

I. INTRODUCTION

THE results of numerous investigations have indicated that silk is probably more readily deteriorated by light than any other natural textile fiber. While the mechanism of the decomposition is not clear, it appears that the rate of decomposition may be considerably altered by pretreatment of the fiber with various chemical reagents. For example, Harris and Jessup[1, 2] found that silk treated with dilute solutions of sodium hydroxide is more stable towards the action of light than untreated silk, whereas the stability of silk treated with dilute solutions of sulfuric acid is greatly decreased. These findings were later substantiated by investigations by Hall[3]. Bonicatti[4], on the other hand, claimed that pretreatment with alkalies has no protective effect but he did observe the photochemical decomposition to be greatly accelerated by treatment of the fiber with dilute solutions of mineral acids. More recently, Weilemann[5] showed that some aliphatic and aromatic amines exhibit a protective action while compounds such as aliphatic amides and amino acids are not effective.

The efficacy of the basic compounds in inhibiting decomposition is of considerable interest, especially since it has recently been found by Gleysteen and Harris[6] that most of the base taken up by silk is bound by the hydroxyl groups of the amino acid tyrosine. It is now shown that the photochemical behavior of silk is associated with the behavior of the tyrosine and that alteration of the tryosine hydroxyl groups by reaction with various reagents is responsible for at least part of the stabilizing effects noted in the past.

II. EXPERIMENTAL PROCEDURE

1. Materials

The silk used in this work was a plain-woven cloth, commercially degummed, having 94 threads per inch. The cloth was first extracted with cold alcohol and with ether and then washed in cold distilled water to remove the organic solvents. This silk is hereafter referred to as *degummed* silk.

The removal of cations from the silk was accomplished either by electrodialysis or by treatment with acid below pH 2.5. This material is hereafter referred to as *electro-*

dialyzed silk or *acid-treated* silk, according to the process by which it was prepared. Purification by electrodialysis has the advantage of eliminating possible secondary reactions which may be produced by treatment of the fibers with acidic solutions.

Methylated samples were prepared using diazomethane according to the procedure recently described by Rutherford, Patterson and Harris[7]. The acidic groups of degummed and of electrodialyzed silk have different rates of reaction with diazomethane, and it was possible to methylate an approximate, predetermined fraction of the carboxylic acid and tyrosine hydroxyl groups. The extent of methylation of each of these groups was directly determined by the procedure previously described[7]. The decrease in tyrosine content is a measure of the hydroxyl groups methylated. The difference between the total methoxyl content and the methoxyl content accounted for by the decrease in tyrosine content is a measure of the carboxyl groups esterified. Samples* I and II were treated for ¼ and for 1½ hours, respectively, is an ethereal solution of diazomethane but were not electrodialyzed or wet out with alcohol before the treatment; sample III was first soaked in 75 per cent alcohol, and then treated for ¼ hour with diazomethane. All of these samples were then electrodialyzed to remove cationic substances. Samples IV and V were electrodialyzed, wet with alcohol, and then treated for ¼ and for 16 hours, respectively, with diazomethane. With the exception of sample V, which lost about 15 per cent in strength, the methylation process had little effect on the strength of the fabric.

2. Methods

The general procedure used in the preparation of the samples for irradiation was as follows: Pieces of the cloth were soaked for 30 minutes in a solution of a given reagent, the ratio of silk to solution being 1 to 100. The greater part of the solution was removed by centrifuging, the cloth being suspended on a wire rack in a centrifuge tube so that practically complete separation of the sample and solution was obtained. When the same piece of cloth was given one or more additional treatments, this same procedure was followed for each treatment. The silk cloth was finally air-dried and then cut into warpwise strips 1¼ inches wide which were then raveled down to 94 warp threads suitable for breaking strength tests. The samples were then exposed to the light for 20 hours. To determine the effect of light alone a second group of samples, treated and prepared in the same way, was kept for 20 hours in the dark under conditions of temperature and humidity approximating those encountered during the light exposure.

The decrease in breaking strength of the cloth was used as a measure of the deterioration of the silk. The tensile strengths were determined on a pendulum type machine, the samples being conditioned and tested in a room maintained at a temperature of 70° C. and a relative humidity of 65 per cent.

*See Table 3.

The source of light was the Eveready accelerated testing unit, type C3A, equipped with Everready Sunshine carbons and Corex D ultraviolet-transmitting glass. The exposures were made for 20 hours at a temperature of 55 to 60° C.†

The cationic contents of the various samples were determined quantitatively by the electrodialytic method of Sookne, Fugitt and Steinhardt[8]. Tyrosine was determined by Lugg's method after an alkaline hydrolysis[9], and methoxyl content by the method of Vieböck and Brecker[10].

III. RESULTS AND DISCUSSION

1. The Effect of Removal of Cations on the Rate of Deterioration

It was observed in preliminary experiments that pretreatment of the silk with practically any basic compound has the effect of reducing the rate of deterioration. This suggested the possibility that the increased stability produced by these substances might result from combination of cations with the acidic groups of the fibers, which are of two types, namely, carboxylic acid and tyrosine hydroxyl groups, and conversely, that the decreased stability observed upon treatment with dilute mineral acids might be due in part to removal of cationic substances from these groups. On the basis of these considerations, it appeared advisable to study the behavior of silk freed from these substances by electrodialysis and by treatment with acid.

The results are shown in Table 1. Electrodialysis of

TABLE 1
Effect of Electrodialysis and of Treatment with Dilute Acids on the Deterioration of Silk Cloth

Sample	*Treatment*	*Cation Content*	*Loss in Breaking Strength on Exposure to Light*
		M. eq./gm.	%
Degummed	None	0.13	32
Electrodialyzed	None	0.01	41
Degummed	H_2SO_4 (pH 2.4)	0.004	41
Degummed	H_2SO_4, followed by water wash.	0.005	41
Degummed	HCl (pH 2.4)	0.002	43
Degummed	HCl, followed by water wash	0.002	43
Degummed	CH_3COOH (pH 2.4)	0.004	43
Degummed	CH_3COOH, followed by water wash.	0.005	41
Electrodialyzed	H_2SO_4 (pH 2.4)	0.004	43

the silk resulted in lowering the cation content from 0.13 to 0.01 milliequivalent per g and at the same time appreciably decreased the stability of the silk to light. Similar results were obtained when the samples were pre-treated with solutions of hydrochloric, sulfuric or acetic acids at pH 2.4**. Thorough washing of the acid-treated samples with distilled water prior to exposure did not alter the stability to light, which indicates that the decrease in stability results from removal of cations and is not caused by the presence of free acid in the exposed samples. It

†This was measured by a mercury thermometer, the bulb of which was covered by a piece of the silk, and placed the same distance from the light as the samples.

**The choice of this pH was based on the titration studies of Gleysteen and Harris[6] in which it was shown that removal of cations from silk becomes appreciable below about pH 3.

(Continued on page 363)

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Annual Meeting and Convention, Carolina Hotel, Pinehurst, N. C., Oct. 31, Nov. 1, 1941.

The Sulfuric Acid and Aluminum Chloride Carbonization Methods for THE DETERMINATION OF WOOL in Mixed Fabrics Compared in an Inter-laboratory Study

LILLIAN E. WEIDENHAMMER
U. S. Bureau of Home Economics

F. A. PRISLEY
Hayward-Schuster Woolen Mills

BERTIL A. RYBERG
American Association of Textile Chemists and Colorists

* * *

FOREWORD

The study described in this paper is a joint contribution of section 5, subcommittee A-3, committee D-13, American Society for Testing Materials and a subcommittee on the Analysis of Fiber Mixtures of the American Association of Textile Chemists and Colorists. Section 5, under the chairmanship of F. A. Prisley, is evaluating the tentative standard methods for the quantitative determination of wool in mixed textiles, in cooperation with a subcommittee of the A.A.T.C.C. under the direction of Bertil A. Ryberg.

At the request of these groups, the Bureau of Home Economics, of the United States Department of Agriculture, distributed fabric samples for analysis to the laboratories of the Botany Worsted Mills, Forstmann Woolen Company, National Bureau of Standards, Sears Roebuck & Company, M. T. Stevens & Sons Company, Pacific Mills, Arlington Mills, American Woolen Company, and the National Research Council of Canada. The data returned by these organizations together with those obtained by analyses made in the Bureau of Home Economics were compared by the statistical procedures reported here.

* * *

ACKNOWLEDGMENTS

Appreciation is expressed to A. E. Brandt, Chief, Conservation Experiment Stations Division, Soil Conservation Service, for assistance with the statistical treatment of this problem. Acknowledgment is also made to W. D. Appel, National Bureau of Standards; A. G. Ashcroft, Alexander Smith and Sons Carpet Company; Margaret B. Hays, Bureau of Home Economics; F. Noechel, Botany Worsted Mills; Laura E. Pratt, Sears Roebuck & Company; W. von Bergen, Forstmann Woolen Company; R. E. Derby, M. T. Stevens & Sons Company; J. N. Dalton, Pacific Mills; H. Christison, Arlington Mills; A. Morrison, American Woolen Company; and P. Larose, National Research Council of Canada, for their co-operation.

* * *

ABSTRACT

In this inter-laboratory study of the sulfuric acid and aluminum chloride methods for the analysis of wool in mixed fabrics, six different fabrics were analyzed by these two methods in a number of cooperating laboratories. First, four wool-rayon mixtures were analyzed in five laboratories. These fabrics had been manufactured for experimental purposes and contained respectively 100 per cent wool, 2/3 wool, 1/3 wool, and 100 per cent rayon.

Since the results were not as conclusive as were desired, further work was planned with the hope that greater standardization of technique and increased representation of laboratories would lead to more definite conclusions. Therefore, two other fabrics were analyzed in ten cooperating laboratories. These fabrics had been manufactured for experimental purposes also, but from blends of wool and cotton carded and combed together. One contained approximately 80 per cent wool, the other 20 per cent wool.

All results, expressed as percentages of wool found, were sent to the Bureau of Home Economics where they were studied. The statistical method of analysis of variance was used in comparing these data. The number of tests necessary for a given precision was calculated. Also details of the techniques employed by the various laboratories were compared.

The variation of five replicates for each method within the individual laboratories proved to be small and fairly uniform, indicating a uniformity of the fabrics in respect to wool content, and also, to some extent, the reliability of the methods.

Unfortunately, except for the all-wool fabric, the exact wool contents were not known so that the accuracy of the methods could not very well be determined. However, the results obtained by each method were in good agreement with the percentages of wool calculated from constituent sulfur content for three of the fabrics. Also, the average percentages for the all-wool fabric obtained by each method compared favorably with the theoretical 100 per cent.

The results obtained by the sulfuric acid method are generally slightly higher than those obtained by the aluminum chloride method. The statistical analysis indicates that the differences, which are all actually very small, are within experimental error in the first part of the work but beyond in the second.

The differences in wool content found in the different laboratories are so small as to be of little commercial consequence even though they are shown by the statistical analysis to be greater than can be attributed to the normal variation due to random sampling.

It would seem from the results of this work that both methods are accurate enough and precise enough to be useful, but the sulfuric acid method is preferable since it is more convenient and less time consuming. Calculations show that for routine testing 2 or 3 samples are probably sufficient for the sulfuric acid method and 3 to 5 for the aluminum chloride method.

INTRODUCTION

THE increasing use of other fibers in admixture with wool in the manufacture of textiles and the recent legislation making mandatory the labeling of wool products increase the importance of the problem of determining the wool content of such mixtures. The present paper describes a comparative study of the aluminum chloride and sulfuric acid methods, under the joint direction of section 5, subcommittee A-3, on wool and its products, committee D-13, American Society for Testing Materials and the subcommittee on Analysis of Fiber Mixtures of the American Asosciation of Textile Chemists and Colorists. These two methods, which had been adopted by A.S.T.M. as tentative standard methods for the analysis of wool-cotton mixtures[1], came under discussion during a meeting of the woolen and worsted fabrics section of subcommittee A-3 in March 1939. It was proposed that similar samples from known wool-rayon fabrics be analyzed by a number of cooperating laboratories, since it was evident from the discussion that a definite need existed for a direct comparison of the methods under routine laboratory conditions, in order to determine which of these two is the better method.

This work was completed during the summer of 1939 and the results were discussed at the October meeting of A. S. T. M. in New York. Since the general consensus of opinion was that the data were not as conclusive as might be desired, it was proposed that further work of a similar nature be undertaken. It was hoped that greater standardization of the techniques involved and increased representation of analysts would furnish data from which more definite conclusions could be drawn.

LITERATURE REVIEW

Methods based on the principle of a dissolving out the cotton and leaving the wool as residue by means of treatment with concentrated sulfuric acid of specified strength, have been discussed by Villavecchia and Cappelli[2], Heermann[3], Schulze[4], Lloyd and Priestley[5], Krais and Markert[6], Griffin[7], and Matthews[8]. The procedure generally described was to treat the sample, from which all non-fibrous material has been removed, with 80 per cent sulfuric acid for from 3 to 6 hours, shaking the mixture vigorously at frequent intervals. Under these conditions the cotton completely dissolved while the wool remained unchanged except for a gain in weight of approximately 1.5 per cent.

Ryberg[9] developed the sulfuric acid method adopted by the American Association of Textile Chemists and Colorists as their standard method, and with which the A. S. T. M. method[1, p. 37] conforms in substance but not in form. He found that, if the sample were pretreated by boiling for from 7 to 10 minutes in 1 per cent sulfuric acid, the cotton dissolved completely within from 10 to 15 minutes upon subsequent treatment with 70 per cent sulfuric acid at 100° F. Wool subjected to these two successive acid treatments did not show an appreciable change in weight. Presumably any increase in weight occurring as a result of treatment with the concentrated acid was counterbalanced by a similar decrease in weight resulting from the pretreatment with diltue acid.

The results obtained by 5 laboratories cooperating under Ryberg's direction in the development of the sulfuric acid method, showed that the method was entirely satisfactory for new wool, but not for reworked wool[9]. The study was made on wool-cotton mixtures, but preliminary observations indicated that silk, regenerated cellulose rayon, and acetate rayon all completely dissolve when treated by this procedure.

Mease and Jessup[10] in a preliminary study of the sulfuric acid method and the aluminum chloride-carbonization method used in some industrial laboratories but hitherto unpublished, found that samples of 100 per cent cleaned wool changed in weight upon treatment with sulfuric acid to a greater extent than did similar samples treated by the different steps of the aluminum chloride method. As a result of these findings they further investigated the aluminum chloride method.

Analyses of prepared wool-cotton mixtures gave percentages of wool content in good agreement with the percentages known to be present. Mease and Jessup concluded that, with the modifications suggested as a result of their work, the aluminum chloride method is convenient and applicable for the accurate determination of wool in mixtures with cotton and regenerated cellulose, but not with cellulose acetate.

MATERIALS AND METHODS

The mixed fabrics used in the first part of this work were manufactured under the direction of the Bureau of Home Economics from known new wool and spun viscose rayon*. Yarns of four different wool contents were spun from carefully weighed wool and rayon, and woven into four different fabrics containing respectively all wool, 2/3

*Appreciation is expressed to the Forstmann Woolen Company for the manufacture of these fabrics.

wool, 1/3 wool, and all rayon. They have been designated as fabrics 1, 2, 3, and 4.

Before they were sampled, the fabrics were degreased; that is, they were extracted with ether for 6 hours, with alcohol for 9 hours, and finally rinsed 10 times in water at approximately 50° C. The extraction was carried out in a special apparatus of the Soxhlet type, so arranged that the freshly distilled solvent was cool by the time it entered the flask containing the fabric.

Five laboratories, designated as A, B, C, D, and E, cooperated. Fifty samples, each weighing from 6 to 8 grams, were cut from a single block of each of the four fabrics, and assigned by means af a randomization scheme to the five laboratories and also to the two methods. Each laboratory, therefore, received 40 samples altogether, 10 from each block, 5 for each of the two methods.

Each laboratory was to follow as closely as possible the directions for the sulfuric acid and aluminum chloride-carbonization methods as outlined in the A. S. T. M. standards on textile materials for 1938[1]. All results were sent to the Bureau of Home Economics where they were tabulated and analyzed. The statistical method of analysis of variance was applied to these data to provide objective tests of the observed differences*. The results for the all-rayon fabrics, 4, were not included in the statistical calculations.

In any study of analytical methods it is, of course, important to know the exact technique employed, especially at those points that might prove to be critical factors in the analysis. Therefore, the cooperating laboratories sent to the Bureau of Home Economics in addition to their numerical results, descriptions of the exact procedures employed, including these points: size of sample; preparation of sample; method of drying sample to constant weight before treatment and residual wool to constant weight after treatment; removal of excess aluminum chloride solution; manipulation to separate carbonized material from wool fibers; preparation of sulfuric acid solution; agitation of samples while immersed in 70 per cent acid; and collection and washing of residual wool, especially pore size and type of filtering apparatus.

The second part of the study differed from the first in several respects. The materials used were two fabrics made specially for Ryberg's work on the sulfuric acid method from blends of cotton and wool carded and combed together[9]. One contained approximately 80 per cent wool, the other approximately 20 per cent wool.

Five other laboratories in addition to the original five cooperated. These new laboratories are designated by the letters F, G, H, J, and K.

Sampling was according to a randomized block and split plot design. From each of five adjoining blocks of fabric, ten samples were cut and assigned at random to the ten laboratories. Each sample was then sub-divided into

*A detailed discussion of the statistical treatment including tables of results will be found in the appendix.

TABLE 1

Percentages of Wool in Four Different Fabrics Reported by Five Cooperating Laboratories Using the Sulfuric Acid and Aluminum Chloride Methods of Analysis

Fabric 1

Sample No.	Lab. A	Lab. B	Lab. C	Lab. D	Lab. E	Mean (All Labs.)	Calc. from Sulfur Content
Sulfuric Acid Method							
1	102.4	99.7	99.9	97.2	99.1		
2	102.8	101.6	99.0	96.4	100.0		
3	102.4	101.7	99.6	97.6	98.0		
4	102.5	101.1	100.5	96.7	99.1		
5	101.9	101.0	99.1	97.5	100.0		
Mean	102.4	101.0	99.6	97.1	99.2	99.9	
Aluminum Chloride Method							
1	101.2	99.7	98.6	97.7	100.3		
2	101.4	100.5	100.0	97.8	100.0		
3	100.5	100.3	99.3	98.0	100.9		
4	100.8	98.7	99.7	98.4	99.6		
5	101.4	100.1	[99.4]*	97.9	100.4		
Mean	101.1	99.9	99.4	98.0	100.2	99.7	
Mean (both methods)	101.7	100.4	99.5	97.5	99.7	99.8	100.0†

Fabric 4

Sulfuric Acid Method							
1	None	0.04	0.04	0.04	0.1		
2	None	.04	.04	.04	.1		
3	None	.06	.06	.03	.0		
4	None	.05	.08	.04	.1		
5	None	.04	.04	.02	.1		
Mean		.05	.05	.03	.08	0.05	
Aluminum Chloride Method							
1	less than 0.05	0.02	0.04	None	0.1		
2	0.05	.02	.02	None	.1		
3	0.05	.04	.04	None	.2		
4	0.05	.06	.01	None	.1		
5	0.05	.05	.01	None	.1		
Mean		.04	.02		.1	0.05	
Mean (both methods)		.04	.04		.1	0.05	0.6

Fabric 2

Sulfuric Acid Method							
1	64.9	63.6	59.3	62.7	62.8		
2	64.6	64.2	62.6	62.6	64.0		
3	65.4	63.9	63.3	62.5	62.8		
4	64.8	63.9	63.1	62.5	63.5		
5	64.8	64.1	63.0	62.2	64.0		
Mean	64.9	63.9	62.3	62.5	63.4	63.4	
Aluminum chloride method							
1	63.4	61.0	60.7	61.6**	60.4		
2	62.2	62.4	60.6	61.7**	61.2		
3	64.8	61.4	60.2	61.8**	60.4		
4	63.4	62.2	62.0	61.6**	59.7		
5	63.2	60.9	62.0	61.9**	60.2		
Mean	63.4	61.6	61.1	61.7	60.4	61.6	
Mean (both methods)	64.2	62.8	61.7	62.1	61.9	62.5	62.9

Fabric 3

	Sulfuric acid method						
1	32.2	30.7	30.1	30.4	30.8		
2	31.7	31.6	30.5	30.6	30.8		
3	31.6	31.0	29.7	30.2	30.9		
4	32.1	31.5	29.6	31.0	30.2		
5	31.3	30.4	30.2	30.8	30.6		
Mean	31.8	31.0	30.0	30.6	30.7	30.8	
	Aluminum chloride method						
1	30.9	30.8	29.8	29.3**	30.7		
2	31.1	30.4	30.4	29.5**	30.2		
3	31.2	31.6	29.0	29.2**	30.1		
4	30.8	29.7	29.3	29.2**	30.1		
5	30.9	30.5	29.3	29.4**	30.4		
Mean	31.0	30.6	29.6	29.3	30.3	30.2	
Mean (both methods)	31.4	30.8	29.8	30.0	30.5	30.5	31.3

*Mean of the 4 other values in this group inserted for purposes of statistical analysis.

†Reference standard for calculations on fabrics 2, 3, and 4.

**Replacement for original samples, which were accidently burned in the course of the analysis.

Fabric 6

	Lab. A		*Lab. B*		*Lab. C*		*Lab. D*	
Block	H_2SO_4	$AlCl_3$	H_2SO_4	$AlCl_3$	H_2SO_4	$AlCl_3$	H_2SO_4	$AlCl_3$
1	21.6	20.7	21.0	20.2	21.6	21.6	21.1	20.3
2	[21.4]*	21.1	21.2	20.9	21.6	21.6	21.1	20.5
3	21.6	20.7	21.0	20.3	21.5	21.8	21.2	20.5
4	21.0	20.7	21.0	20.6	20.8	20.6	20.8	20.3
5	21.3	20.1	20.8	20.5	20.6	20.4	21.0	20.8
Mean	21.4	20.7	21.0	20.5	21.2	21.2	21.0	20.5
Mean (both methods)	21.0		20.8		21.2		20.8	

	Lab. E		*Lab. F*		*Lab. G*		*Lab. H*	
Block	H_2SO_4	$AlCl_3$	H_2SO_4	$AlCl_3$	H_2SO_4	$AlCl_3$	H_2SO_4	$AlCl_3$
1	21.1	20.7	20.8	21.0	20.8	21.0	22.2	20.9
2	21.3	20.5	20.2	21.1	20.8	20.7	21.8	20.9
3	21.4	20.3	21.0	21.4	20.4	20.5	21.2	20.4
4	21.5	20.9	21.1	20.3	20.8	20.7	21.6	20.5
5	21.0	20.4	20.7	21.3	20.3	20.5	21.6	20.3
Mean	21.3	20.6	20.8	21.0	20.6	20.7	21.7	20.6
Mean (both methods)	21.0		20.9		20.6		21.2	

	Lab. J		*Lab. K*		*Means for Blocks and Methods*			
Block	H_2SO_4	$AlCl_3$	H_2SO_4	$AlCl_3$	*Block*	H_2SO_4	$AlCl_3$	*Mean*
1	20.1	22.0	21.7	20.3	1	21.2	20.9	21.0
2	20.6	21.6	21.6	20.2	2	21.2	20.9	21.0
3	20.7	22.3	21.1	20.0	3	21.1	20.8	21.0
4	20.3	22.0	21.4	20.3	4	21.0	20.7	20.8
5	20.1	22.0	21.2	19.9	5	20.9	20.6	20.8
Mean	20.4	22.0	21.4	20.1	Mean	21.1	20.8	21.0
Mean (both methods)	21.2		20.8					

*Mean of the 4 other values in this group inserted for statistical purposes.

TABLE 2

Percentages of Wool in Two Different Fabrics Reported by Ten Cooperating Laboratories Using the Sulfuric Acid and Aluminum Chloride Methods of Analysis

Fabric 5

	Lab. A		*Lab. B*		*Lab. C*		*Lab. D*	
Block	H_2SO_4	$AlCl_3$	H_2SO_4	$AlCl_3$	H_2SO_4	$AlCl_3$	H_2SO_4	$AlCl_3$
1		77.4	78.5	76.9	76.3	75.9	78.1	79.3
2		77.0	78.3	76.6	75.9	75.6	78.5	76.0
3		77.0	78.2	76.8	78.4	78.2	78.8	75.5
4		75.8	78.5	76.9	76.0	75.6	78.0	77.4
5		75.4	78.5	76.1	77.5	78.0	78.0	75.5
Mean		76.5	78.4	76.7	76.8	76.7	78.3	76.7
Mean (both methods)	76.5		77.6		76.8		77.5	

	Lab. E		*Lab. F*		*Lab. G*		*Lab. H*	
Block	H_2SO_4	$AlCl_3$	H_2SO_4	$AlCl_3$	H_2SO_4	$AlCl_3$	H_2SO_4	$AlCl_3$
1	79.8	77.3	78.5	77.4	78.0	77.1	78.0	79.3
2	78.5	77.8	78.6	76.6	78.4	77.1	79.0	79.0
3	79.5	76.6	79.4	75.8	78.4	77.0	78.9	80.0
4	79.4	77.7	78.2	77.7	78.6	77.1	79.8	78.5
5	78.7	77.7	78.9	78.3	78.4	77.1	79.2	79.5
Mean	79.2	77.4	78.7	77.2	78.4	77.1	79.0	79.3
Mean (both methods)	78.3		78.0		77.8		79.2	

	Lab. J		*Lab. K*		*Means for Blocks and Methods*			
Block	H_2SO_4	$AlCl_3$	H_2SO_4	$AlCl_3$	*Block*	H_2SO_4	$AlCl_3$	*Mean*
1	76.4	79.0	79.0	67.0	1	78.1	76.7	77.4
2	74.6	78.9	79.1	73.1	2	77.9	76.8	77.4
3	74.8	79.4	78.7	74.9	3	78.3	77.1	77.7
4	75.7	79.3	78.4	73.2	4	78.1	76.9	77.5
5	74.2	78.6	78.5	77.2	5	78.0	77.3	77.6
Mean	75.1	79.0	78.7	73.1	Mean	78.1	77.0	77.5
Mean (both methods)	77.1		75.9					

two parts which were assigned, also at random, to the two methods.

The fabrics were not degreased before sampling as was done previously. The samples sent out to each laboratory were accompanied by the following detailed directions. Degreasing was included as it was felt that this was a part of the analytical procedure and should therefore be carried out in the same laboratory where the determination of wool content was made.

Preparation of Materials:—Samples for both methods should be prepared by extracting with carbon tetrachloride. From results of analyses of cotton fabrics for total sizing made in the laboratories of the Bureau of Home Economics, it would seem that the carbon tetrachloride extraction can be carried out as satisfactorily by the more convenient single 2-hour immersion in the solvent contained in an ordinary beaker as by extracting for 2 hours in a Soxhlet apparatus siphoning over at least six times. After the treatment with carbon tetrachloride, the sample is air dried and thoroughly rinsed three times, with hot distilled water at 50° C.

Sulfuric Acid Method:—Approximately two grams of the above prepared sample are dried to constant weight[1]. The weighed sample is then immersed in 200 ml., or 100 X

the weight of the sample, of a boiling 1 per cent solution of sulfuric acid (this solution which is 1 per cent by weight and contains 1 gram of H_2SO_4 in 100 grams of solution, can be prepared by adding 5.9 ml. of concentrated 93 per cent H_2SO_4 to 990 ml. of water) for 7 to 10 minutes.

The sample is then transferred to a Buchner or Hirsch funnel, 45 mm. in diameter, and the excess acid solution removed by suction. It is allowed to cool, and then held over a 400 ml. beaker and carefully cut into strips and shreds approximately ⅛ inch by 1 inch. Experience has shown that it is not necessary to shred the sample for the first treatment. Shredding while still wet prevents any loss of fiber and so prepares the sample that it insures complete removal of the cellulosic material by the 70 per cent acid.

Two hundred ml. of 70 ± 1 per cent sulfuric acid solution (A 70 per cent solution of sulfuric acid by weight, containing 70 grams of H_2SO_4 in 100 grams of solution, is equivalent to 55° Baumé, 123° Twaddel or 1.620 specific gravity at 60° F., and is easily prepared by carefully pouring 800 ml. of commercial oil of vitriol of 66° Baumé into 484 ml. of distilled or soft water stirring and cooling to the desired temperature. This 70 per cent acid is available commercially as 55° Baumé sulfuric acid and may be purchased from any of the chemical supply houses at a very reasonable price) at 100° F. are added and the whole allowed to stand 15 minutes with frequent stirring.

A 1-liter beaker is partly filled with 600 ml. of cold water and at the expiration of the 15 minutes the 70 per cent acid solution containing the sample is cautiously but quickly poured into it. The 400 ml. beaker is rinsed with two 50 ml. portions of water which are added to the 1-liter beaker. The residue is then recovered by filtering the above solution in the Buchner or Hirsch funnel through a disc of bleached, unstarched 80/80 cotton print cloth using suction. The sample is rinsed with several portions of cold water, sucked reasonably dry and the whole pad, including disc and fibers, is then carefully lifted out of the funnel by means of tweezers and dropped into 200 ml. of a cold 2-per cent solution of sodium bicarbonate and neutralized for 5 minutes. It is again collected on the same filter disc in the funnel, rinsed with several portions of cold water, sucked reasonably dry, and the disc lifted out and placed in a warm sheltered spot to dry. The object of this preliminary drying is to facilitate the transfer of the fiber mass from the cotton filter disc to the weighing bottle. When dry, the recovered fiber mass is carefully transferred to a weighing bottle, placed in the drying oven and dried to constant weight. Results should be calculated on an Oven Dry Basis.

Aluminum Chloride Method:—The sample, approximately 2 grams in weight, after the carbon tetrachloride extraction and hot water rinsing, is cut into 1 to 2 inch squares and unraveled, and then dried to constant weight by heating in weighing bottles in an oven at 105°-110° C. The dry, weighed sample is then immersed for 10 minutes in approximately 100 ml. of a boiling aluminum chloride solution containing 5 g. of $AlCl_3$, or 9 g. of the hydrated salt as $AlCl_3.6H_2O$, per 100 ml. of water. It is removed from the solution and after allowing the excess liquid to drain off without squeezing the specimen, the mass is spread on a watch glass and heated in an oven at 105° to 110° C. until the cotton has become brown in color and brittle. Usually two hours are sufficient.

The specimen is rubbed gently against a 100-mesh screen, with just enough pressure to pulverize the charred cellulose and with occasional tapping of the screen to shake the powdered material through it. The screenings are collected on a clean dry paper. Re-drying of the specimen for short intervals may be necessary to remove all charred cellulose, as aluminum chloride is hygroscopic so that on damp days the carbonized specimen may take up sufficient moisture from the air to cause it to become sticky before the cellulose has been completely rubbed out.

The residual wool fibers are placed in a suitable container preferably a 250 ml. beaker. The material which passed through is returned to the screen and rinsed with water to recover any wool fibers that had passed through the carbonized material. The wool is agitated with 100 ml. of dilute hydrochloric acid solution prepared by diluting 1 part by volume of the concentrated laboratory reagent with 9 parts of distilled water. The mass is then collected as a pad by filtering in a Buchner or Hirsch funnel through a disc of bleached, unstarched 80/80 cotton print cloth using suction. It is washed with distilled water until free of chlorides and dried in the air. When dry the wool is removed from the disc and dried to constant weight as before. The percentage of wool is calculated from this dry weight and the original dry weight of the sample.

The above procedures are, essentially, adaptations for this particular study of a revision of the sulfuric acid method prepared by Ryberg, and of the aluminum chloride method as outlined in A.S.T.M. standards on textile materials for 1939[11].

The number of replicate samples necessary for a given precision in analyzing mixed fabrics by either of these two methods was calculated following the "proposed recommended practice for calculating number of tests to be specified in determining average quality of a textile material" appearing in the 1940 edition of A.S.T.M. standards[12].

DISCUSSION OF RESULTS

The accuracy of the sulfuric acid and aluminum chloride methods could not be determined with any degree of certainty from these studies since the exact wool contents of the fabrics used were not known. The fact that the average percentages, 62.5 and 30.5, for fabrics 2 and 3 are so much lower than the theoretical values, 66.7 and 33.3 per cent, leads to the conclusion that either both methods are inaccurate, or manufacturing yarns from weighed quantities of different fibrous materials, such as wool and rayon, does not yield sufficiently exact proportions in the final fabrics to serve as a check on methods of analysis.

The average of 99.8 per cent for the all-wool fabric is in excellent agreement with the theoretical 100 per cent. Fabrics 5 and 6, approximately 80 per cent and 20 per cent wool respectively, were found to contain 78.1 and 21.1 per cent by the sulfuric acid method and 77.0 and 20.8 per cent by the aluminum chloride method, or 77.5 and 21.0 per cent when the results for both methods are averaged.

That traces of wool were found in the supposedly all-rayon fabric shows to some extent the exactness of the separation in both methods. These traces were in all probability wool fibers and not undissolved rayon. Those obtained in the Bureau of Home Economics laboratory when examined under the microscope proved to be wool. Their presence is readily accounted for, inasmuch as the mill where this fabric was manufactured usually handles nothing but wool. It is very likely that bits of wool still clinging to the machinery were caught up during the manufacturing of the spun rayon fabrics.

Another check on the accuracy of the methods of analysis can be obtained by calculating the percentage of wool from the constituent sulfur content. It is necessary, of course, to have a comparable all-wool fabric to use as a reference standard. For the wool-rayon fabrics, fabric 1 can be so used. On the basis of sulfur content the percentage of wool in fabrics 2, 3, and 4 were calculated, assuming fabric 1 to contain 100 per cent wool.

The values 62.9, 31.3, and 0.6 per cent so obtained are in good agreement with the averages of 62.5, 30.5, and 0.05 per cent for both methods, and also with 61.6, 30.2, and 0.05 per cent for the aluminum chloride method, and 63.4, 30.8, and 0.05 per cent for the sulfuric acid method. It is known that viscose rayon contains small amounts of sulfur, retained from the xanthate stage of the manufacturing process, which would account for the high wool percentage for fabrics 3 and 4, as calculated from sulfur content.

Both uniformity of the fabric and reliability of the methods are indicated by the small differences in percentage of wool content found for replicate samples.

The results obtained by the sulfuric acid method are slightly higher generally than those by the aluminum chloride method. That this difference between methods is small is shown by the differences 0.2, 1.8, 0.6, 1.1, and 0.3 between the averages for each method for fabrics 1, 2, 3, 5, and 6, respectively. Statistical analysis indicates that in the first part of the study the differences between methods are within experimental error, in the second part they were not.

The differences in wool content found in the different laboratories, although significant, that is, greater than can be attributed to normal variation resulting from random sampling, are so small as to be of little commercial consequence.

A survey of the details of the procedure followed in the different laboratories failed to show any close correlation between departures in technique and variability of results between laboratories. Both methods apparently allow considerable latitude in technique before results are greatly affected. For example, in the aluminum chloride method, the separation of the carbonized material from the wool was carried out in one laboratory entirely by screening in the dry state, while in another laboratory this was done by first rubbing the carbonized sample between the hands over the screen and then washing thoroughly. The average wool content found in the first laboratory was 0.7 per cent higher than that reported by the second for fabric 5, and 0.1 per cent higher for fabric 6.

In several of the laboratories changes in technique for the sulfuric acid method were made within the laboratory itself without affecting results very greatly. In the light of some of this work the procedure for this method has been revised further.

In one instance, however, departure from the outlined technique is a possible explanation for results completely at variance with those obtained in the other laboratories. The data reported by laboratory J showed a markedly higher wool content for samples analyzed by the aluminum chloride method than for those analyzed by the sulfuric acid method, whereas in every other instance where a significant difference between the two methods was found it was always the sulfuric acid that gave the higher result. It was suggested after further investigation in this laboratory that the high results might have been caused by incomplete wetting of the fibers by the aluminum chloride solution, as the sample was not unraveled before treatment with the aluminum chloride.

It is interesting to note, too, that with both methods increased familiarity with the technique apparently tends to minimize whatever factors are causing inter-laboratory discrepancies and to produce greater uniformity in results. For example, the results obtained in the second study by the five laboratories that cooperated in the first were in good agreement. Statistical analysis showed that the differences were in this instance no greater than could be attributed to normal variation. Also, laboratories F, G, H, and K had previously cooperated in a study of the sulfuric acid method. The average percentages found by these laboratories using the sulfuric acid method, 78.4, 78.7, 78.7 and 79.0, are in excellent agreement, whereas those obtained by the aluminum chloride method, 73.1, 77.1, 77.2 and 79.3, are more variable.

This work leads to the conclusion that both methods are accurate enough and precise enough to be used. However, analysts quite generally agree that, from the standpoint of time consumed and ease of handling, the sulfuric acid method is greatly to be preferred.

At the October, 1940, meeting, section 5 of A. S. T. M. Sub-committee A-3, on the basis of this work and the opinions expressed at that time, voted to recommend that the aluminum chloride method be deleted from the D-13 manual and the sulfuric acid method be accepted as the tentative standard for analysis of wool.

For any standard method it is of considerable importance to know the number of replicate samples that should be taken in order that there be a high degree of probability

TABLE 3
Number of Tests for the Sulfuric Acid and Aluminum Chloride Methods for the Analysis of Wool in Mixed Fabrics

Sulfuric acid method					*Aluminum chloride method*				
Fabric	*No. of Subgroups*	*Grand Mean,* $\bar{\bar{X}}$	*Universe Standard Deviation,* σ'	*Universe Coefficient of Variation,* v'	*Fabric*	*No. of Subgroups*	*Grand Mean,* $\bar{\bar{X}}$	*Universe Standard Deviation,* σ'	*Universe Coefficient of Variation,* v'
1	5	99.9	0.655	0.656	1	5	99.7	0.508	0.510
2	4*	63.7	.351	.551	2	5	61.6	.666	1.081*
3	5	30.8	.394	1.279	3	5	30.2	.381	1.262
5	9	78.1	.551	.706†	4	9‡	77.4	.774	1.000
6	10	21.1	.290	1.374**	5	10	20.8	.327	1.572†

High wool content

Allowable Random Sampling Error, E, Per Cent	*Number of Tests Where P Equals* 0.90	0.95	0.99
0.5	6	8	14
1	2	2	4
1.5	2	2	3
2	1	1	1
2.5	1	1	1

High wool content

Allowable Random Sampling Error, E, Per Cent	*Number of Tests Where P Equals* 0.90	0.95	0.99
0.5	13	18	31
1	4	5	8
1.5	3	4	7
2	1	2	2
2.5	1	1	2

Low wool content

Allowable Random Sampling Error, E, Per Cent	*Number of Tests Where P Equals* 0.90	0.95	0.99
0.5	21	29	50
1	6	8	13
1.5	5	6	10
2	2	2	4
2.5	1	2	2

Low wool content

Allowable Random Sampling Error, E, Per Cent	*Number of Tests Where P Equals* 0.90	0.95	0.99
0.5	27	38	66
1	7	10	17
1.5	6	8	14
2	2	3	5
2.5	2	2	3

*Data from laboratory C omitted because σ for these data is beyond control limits.
†Selected coefficients of variation for fabrics of high wool content.
**Selected coefficients of variation for fabrics of low wool content.
‡Data from laboratory K omitted because σ for these data is beyond control limits.

that the average values obtained from repeated sets of determinations will fall within a given close range. This number of tests can be calculated for a given probability from a predetermined allowable random sampling error and the observed variability inherent in the method shown by the differences between values for samples treated alike.

Such calculations were made for both methods for each of the experimental fabrics except fabric 4, and also for several probabilities and several random sampling errors. The results indicate that it is possible to generalize the conclusions by grouping these mixed fabrics into two groups, one containing a high wool content of approximately 50 to 100 per cent, and the second containing a low wool content of approximately 0 to 50 per cent. On this basis, fabrics 1, 2, and 5 were considered representative of the first group, and fabrics 3 and 6 of the second. However, instead of averaging the coefficients of variation in each group and calculating the number of tests from that, the highest one was selected in order to insure a large enough number of tests. Fabrics 5 and 6 showed the highest coefficients of variation with the sulfuric acid method; fabrics 2 and 6 with the aluminum chloride. The number of tests for these fabrics for the various probabilities and precisions are shown in Table 3.

It is apparent from this table that in analyzing mixed fabrics by the sulfuric acid method two samples are sufficient for a precision of one per cent 95 times out of 100 for materials of high wool content, and for a precision of two per cent for those of low content. With the aluminum chloride method five and three samples are necessary respectively for the same probability and precisions. For example, the chances are 95 in 100 that the average percentage of wool for two samples from fabric 5, analyzed by the sulfuric acid method will lie within the range 78.1 $\pm$ 0.8; and within 21.1 $\pm$ 0.4 for two samples from fabric 6; while for these same fabrics analyzed by the aluminum chloride method five samples are required for a 95 per cent probability that the average will lie within the range 77.4 $\pm$ 0.8 for fabric 5, and 20.8 $\pm$ 0.4 for fabric 6.

A study of the apparent variability inherent in these methods indicates that 1 per cent for fabrics of high wool content and 2 per cent for those of low content are probably the allowable random sampling errors that should be selected. Manufacturers have found that it is more difficult to obtain a uniform mixture of cotton and wool fiber when the wool content is low than when it is high. This would account for the difference in precision in the analysis of these fabrics. A probability of 95 per cent is generally considered satisfactory for most test methods. It may be concluded from this, therefore, that for purposes of routine testing 2 to 3 samples are sufficient for the sulfuric acid method and 3 to 5 for the aluminum chloride.

From the above it would seem that results obtained by the aluminum chloride method are somewhat more variable

than those obtained by the sulfuric acid method. From a survey of the standard deviations for each method, however, it would seem that the two methods do not differ greatly in this respect. This discrepancy can probably be explained by the fact that in the attempt to make the calculated number of tests more general the extremes of variation were chosen and these extremes were greater for the aluminum chloride than for the sulfuric acid method. This, in turn, is undoubtedly due to the fact that the aluminum chloride method seems to present more experimental difficulties. It does not lend itself as readily to standardization for routine work as does the sulfuric acid method, and, therefore, even though it is as reliable, inherently, there are more instances of erratic results than is the case with the sulfuric acid method, especially whenever the operator is unfamiliar with the technique. This again leads to the conclusion that the sulfuric acid method is to be preferred as a standard method.

CONCLUSIONS

The results of this work lead to the conclusion that both methods are accurate enough and precise enough to be useful, but because it is more convenient and less time consuming the sulfuric acid method is preferable. The results of the calculations of number of tests that should be made indicate that for ordinary routine testing 2 to 3 samples are probably sufficient for the sulfuric acid method and 3 to 5 for the aluminum chloride method.

APPENDIX—STATISTICAL TREATMENT

In both parts of this study five samples from each fabric were analyzed by each method in each cooperating laboratory. In the first part the samples were taken from a large single block of fabric and assigned at random to both the laboratories and the methods by using a set of random numbers obtained from tables[13]. This design leads to the analyses summarized in Table 4.

A test of homogeneity of the contributions to variance from each set of five replicates by the chi-square method[14] showed that the results from laboratory C for the analysis of fabric 2 by the sulfuric acid method contributed so much more to the variance than any of the other sets of five replicates that this group of data ought to be excluded from any combined analysis of variance.

Moreover, in laboratory D, the original samples from fabrics 2 and 3 were accidentally burned in the aluminum chloride process and were replaced by new ones from the same fabrics, but cut from an entirely different section than the block sampled here. These values, although worth examining, were not included in the combined analysis.

Four analyses of variance were made therefore. For analysis 1, the data on fabric 1 were used. Included in analysis 2 were fabrics 1 and 3 and laboratories A, B, C, and E; in analysis 3, fabrics 1, 2, and 3 and laboratories A, B, and E; and in analysis 4, fabrics 1 and 3 and laboratories A, B, and E.

The sums of squares for the comparisons given in the table were obtained by the method of making individual comparisons for each degree of freedom and then totaling in appropriate groupings the individual sums of squares so obtained[14].

The appropriate estimates of error for the tests of significance made in these analyses are the mean squares for the inter-action between the factors studied. For analysis 1 the interaction is "method X laboratory"; for the other three analyses all the interactions with "fabric" are combined to obtain the error term, that is, "fabric X method," "fabric X laboratory" and "fabric X method X laboratory."

The results of these analyses show that, except in the case of analysis 3, the differences between the percentages of wool found by the two methods are not significant, which means that these differences are no larger than might have been encountered if only one method had been used throughout. The results of analysis 3, in which fabrics 1, 2, and 3 and laboratories A, B, and E were included, indicate that the methods did differ significantly. The large error mean square, 3.18, as compared with 0.93 in analysis 2, which included laboratory C and excluded fabric 2, would indicate that fabric 2 was a disturbing factor. This is further shown by the fact that the results of analysis 4, in which fabrics 1 and 3 and laboratories A, B, and E were included, are in line with those of analysis 2.

TABLE 4
Analyses of Variance of Wool Content for Three Fabrics Reported by Five Cooperating Laboratories Using Two Different Methods

	Analysis 1, Including Fabric 1 and Labs. A, B, C, D, E.		*Analysis 2, Including Fabrics 1, 3 and Labs A, B, C, E.*		*Analysis 3, Including Fabrics 1, 2, 3 and Labs. A, B, E.*		*Analysis 4, Including Fabrics 1, 3 and Labs. A, B, E.*	
Comparison	*D/F*	*Mean Square*	*D/F*	*Mean Square*	*D/F*	*Mean Square*	*D/F*	*Mean Square*
Between fabrics			1	97270	2	36560	1	72960
Between methods	1	0.35	1	4.47	1	27.78*	1	4.00
Between laboratories	4	23.55**[1]	3	13.36**	2	22.52*	2	10.71*
Method x laboratory	4	3.01[2]	3	1.85	2	.566	2	2.72
Fabric x method Fabric x laboratory Fabric x method x laboratory			7	.93[2]	10	3.18[2]	5	1.28[2]
Between replicates	40	.332	64	.28	72	.306	48	.281
Total	49		79		89		59	

[1]One asterisk indicates a significant variation; two asterisks a highly significant variation.
[2]Error term.

TABLE 5
Analyses of Variance of Wool Content for Two Fabrics Reported by Ten Cooperating Laboratories Using Two Different Methods

Comparison	*Analysis for Fabric 5 and Labs. B, C, D, E.* D/F	Mean Square	*Analysis for Fabric 6 and Labs. A, B, C, D, E.* D/F	Mean Square	*Analysis for Fabric 5 and Labs. B, C, D, E, F, G, H, J.* D/F	Mean Square	*Analysis for Fabric 6 and Labs. A, B, C, D, E, F, G, H, J, K.* D/F	Mean Square
Whole plots								
Between blocks	4	0.512	4	0.298	4	0.429	4	0.321
Between laboratories	3	4.06	4	.370	7	5.40**	9	.395**
Error (a) (block x laboratory)	12	1.21	16	.153	28	.691	36	.125
Split plots								
Between methods	1	16.90**[1]	1	3.12**	1	4.66**	1	2.10**
Method x laboratory	3	1.47	4	.202**	7	9.29**	9	1.72**
Error (b)								
Block x method Block x method x laboratory	16	.564	20	.0382	32	.497	40	.0518
Total	39		49		79		99	

[1]One asterisk indicates a significant variation; two asterisks a highly significant variation.

Both uniformity of the fabric and reliability of the methods are indicated by the small differences in percentage of wool content found for replicate samples. It will be noted that the mean square for the between replicates comparison is much smaller than the mean square for interaction involving fabrics.

Lack of agreement between laboratories is clearly shown since according to all four analyses of variance the laboratories differed significantly or highly significantly. "Significant" and "highly significant" means that the probabilities are only 5 per cent and 1 per cent respectively that these interlaboratory differences would have been obtained had all samples been analyzed in the same laboratory. This disparity cannot be attributed to any one laboratory. It is true that the average percentages reported by laboratory A are consistently higher, but the results obtained by the other four laboratories are not in close agreement.

In the second part of the study the fabrics were marked off into adjoining blocks of ten samples each. The ten samples in turn were assigned at random to the ten laboratories, and, further, were subdivided into two parts, one for each method. In the analysis for this design separate estimates of error can be found for the comparisons involving the whole plots and those involving the split plots.

Since one set of data was missing and another set showed a lack of control known to be due to experimental difficulties and therefore could not be used for purposes of statistical analysis, separate analyses were made for each fabric in order to utilize all of the available data. Also, data from the five laboratories that had participated in the first study were analyzed separately in order to determine the effect of previous experience.

The sums of squares were calculated in the ordinary manner[14]. However, those for the interactions were obtained directly and not by subtractions involving sums of squares already obtained.

These results, given in Table 5, show a significant difference between the methods. This means that the sulfuric acid method yielding higher results than the aluminum chloride method, is in all probability attributable to an inherent difference in the methods and not to accident.

Furthermore, the "method X laboratory" interaction is significant in three of the four instances. This indicates that the differences between methods were not the same in the different laboratories, or vice versa, that any interlaboratory variations were not the same for the two methods.

Lack of agreement between the laboratories is again shown but only where the results from eight or ten laboratories are compared. In the analyses involving only those laboratories that had cooperated in the first part of the study the difference between results from the various laboratories is not significant.

Again, there is no instance of a significant difference between replicates, showing that these fabrics, too, are fairly uniform in respect to their wool content.

The number of tests that should be made in analyzing by these two methods such mixed fabrics as were used in this work were calculated following the procedure proposed by Committee D-13 of the American Society for Testing Materials[12]. The results are shown in Table 3. The cooperating laboratories were considered as subgroups rather than sources. Case 1 applies since these sub-groups each contain five tests. The correction factor, C_2, in this case is 0.8407.

From a study of the coefficients of variation obtained for fabrics 1, 2, 3, 5, and 6 it seemed reasonable to divide these roughly into two groups of sources, namely, fabrics of a high wool content, approximately 50 to 100 per cent, and those of low wool content, approximately 0 to 50 per cent. Fabrics 1, 2, and 5 are, therefore, sources in the first group, and fabrics 3 and 6 in the second. The coefficient of variation selected for calculating the number of tests was the highest universe coefficient of variation in each group in order to increase the assurance that the numbers of samples calculated are large enough.

Any choice of the number of samples to be taken for purposes of analyzing a mixed fabric for wool content by

either of these methods must be made on the basis of judgment based in turn upon experience and the purposes of the analysis. The following conclusion can, however, be drawn: When the coefficient of variation of wool content of mixed fabrics as analyzed by the sulfuric acid method is 0.706 per cent, which, it would seem from results of this work, is not generally exceeded for mixed fabrics containing more than 50 per cent of wool, the random sampling error of the average of 2 tests should not be greater than 1 per cent of the universe average wool content approximately 95 times in 100. Similar conclusions can be stated for the other three cases, namely, fabrics of low wool content analyzed by the sulfuric acid method, and fabrics of both high and low content analyzed by the aluminum chloride method.

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ONE HUNDRED AND TWENTY-SIXTH MEETING OF THE RESEARCH COMMITTEE

THE 126th meeting of the Research Committee followed the Council meeting in New York on Saturday, June 14, 1941. Present were Chairman Louis A. Olney presiding, P. J. Ariente, G. L. Baxter, J. R. Bonnar, W. H. Cady, H. Christison, C. Z. Draves, W. Ebersold, E. Freedman, A. H. Gaede, R. Hart, A. E. Hirst, D. P. Knowland, A. R. Macormac, O. F. Marks, W. R. Moorhouse, G. A. Moran, C. L. Nutting, R. W. Philip, B. A. Ryberg, R. E. Rupp, C. H. A. Schmitt, C. A. Seibert, T. R. Smith, H. W. Stiegler, B. Verity, W. v. Bergen, P. J. Wood, and H. C. Chapin, Secretary.

Reporting for the Committee on Fastness to Dry Cleaning, Mr. Seibert stated that they were about to send out another questionnaire to clear up points raised by an earlier one. Study of the Fastness to Chlorine test he said was continuing with view to its adaptation to a wide range of laboratory temperatures in various parts of the country, and adjustment of commercial hypochlorites to conform with test specifications. Differences among these, in their action on dyes, even after buffering to a standard pH, indicated possibility of improvement in some of these preparations through conformity to certain manufacturing specifications.

Mr. Bonnar reported increasing use of the test for moth repellency, evident from inquiries and sale of standard cloth, and progress toward unification of the test methods of various organizations. A revised method of testing gas fading of dyed acetate rayon, presented by Mr. Cady for publication in the 1941 Year Book, was accepted as a tentative standard method. A modification of Dr. Draves' Wetting Out Test, proposed by him for evaluation of weting out agents in mercerizing liquors, was accepted as a tentative standard method. For the Light Fastness Committee Mr. Cady reported that the four intermediate British and German standards were not quite satisfactory in gradation for Association use, and that attempts were being made to adapt them by slight modifications. After an extended discussion initiated by Mr. Moran it was decided that the tests for "Fastness to Light of Dyestuffs on All Textiles," and "Fastness to Light of Dyed Textiles" were the same in fundamental purpose, and should be combined into one test in the 1941 Year Book. It was voted that the tentative optional method of testing fastness to perspiration of viscose and acetate linings and dress goods, through use of heated oven, be included in the official method as optional for all fabrics. The susceptibility of certain dyes to color change through joint effect of perspiration residues and chlorinated hydrocarbons was discussed, with decision that a spotting test with dilute hydrochloric acid be added to the perspiration test to catch these peculiar cases.

A letter from Dr. A. C. Goodings of the Ontario Research Foundation suggested a committee to deal with mildew and bacterial damage, and the Chairman was authorized to appoint such a committee. A letter from Duncan Ferguson recommending standard methods of color inspection was referred to the Committee on Color. Mr. Ariente was made Chairman of a committee to deal with fastness of dyed rayon to washing, and Mr. Marks Co-Chairman to deal with shrinkage for the same. Consideration was given to the appointment of a committee to confer with the Philadelphia Quartermaster Depot on olive drab dyeing of wool, as voted by the Council.

Respectfully submitted,

HAROLD C. CHAPIN, *Secretary.*

Bleaching Cotton Goods with TEXTONE Activated with Hypochlorite*

G. P. VINCENT, A. L. DUBEAU and J. F. SYNAN

Development Department, Mathieson Alkali Works (Inc.)

WHENEVER cotton fibers are acted upon by either calcium hypochlorite or sodium hypochlorite, a compound known as oxycellulose tends to form.

This degradation product is objectionable in yarns or cloth because it causes tendering of the fiber, the development of yellowish or brownish stains on storage, and uneven dyeing.

In bleaching cotton goods with hypochlorite, the amount of oxycellulose formed can be kept down to a minimum by a careful chemical control of every step of the process, and especially, by stopping the bleaching action before it has gone beyond a certain point. However, it is ordinarily impossible to prevent completely the formation of oxycellulose.

Research work carried out during the past two years by the Research and Development Department of the Mathieson Alkali Works, Inc., showed that all danger of forming oxycellulose can be avoided by bleaching the goods by means of sodium chlorite ($NaClO_2$) in an acid solution instead of using hypochlorite in an alkaline solution.

Chlorite, which is supplied in a form suitable for use in textile finishing under the name of Textone, has sufficient oxidizing power to destroy all the coloring matter associated with the cotton fiber but it is not powerful enough to attack the fibers themselves. Hence, it can be used safely without careful control of time, temperature and concentration, producing better and more permanent whites without loss of tensile strength.

Where arrangements are made to carry out the use of Textone in an acid solution, bleached good of a high quality can be produced at a low cost, but the process requires radical changes in bleaching methods and, perhaps, the use of special equipment.

*Presented at meeting, Rhode Island Section, May 23, 1941.

SUMMARY

The results of laboratory experiments and full-scale plant trials indicate that a mixture of sodium hypochlorite and sodium chlorite (Textone) is superior for bleaching cotton goods to either straight calcium hypochlorite or straight sodium hypochlorite, because this mixture does not attack the fibers to form oxycellulose.

Tests show that goods bleached by Textone activated with hypochlorite have greater fiber strength than those bleached with ordinary chemic. In addition, the whiteness is more permanent.

The Textone-hypochlorite solution is used in the same way as ordinary chemic is used, and the same equipment can be employed. The solution does not attack cotton fiber so that careful control of time, temperature, and concentration to prevent injury is not required. Its bleaching power is approximately equal to that of chemic having twice as much available chlorine.

Directions for preparing activated Textone solutions for bleaching various kinds of cotton goods are given.

However, further research has shown that excellent results can be secured, without altering ordinary bleaching procedures in any important particular, if a mixture of Textone and hypochlorite in an alkaline solution is used in the place of straight hypochlorite. This is particularly true in continuous bleaching employing impregnation and stacking.

The bleaching action of this mixture is characteristic of Textone and not of hypochlorite. Chemically, the reason probably is that chlorine dioxide (ClO_2), which bleaches without cellulose degradation, is formed by the reaction between Textone and hypochlorite.

LABORATORY WORK

In order to determine the strength, proportions, and pH of the Textone-hypochlorite mixture best suited for use in bleaching cotton the following experiments were carried out.

Strength and Ratio Tests

In these experiments, the cloth used was a slub yarn broadcloth, which had been singed, mercerized, and kier-boiled.

The regular procedure for bleaching this cloth was:

Impregnate with 3 gpl. (grams per liter) hypochlorite, cold; stack 5 to 6 hours; wash; anti-chlor; wash; dry.

In bleaching this cloth in the laboratory, two series of tests were made: (1) with cloth not soured after kiering, and (2) cloth that was soured after kiering. In each series the bleaching procedure was as follows:

Impregnate with one of the solutions given below, used cold; stack 6 hours; wash; anti-chlor, with SO_2 water, cold; wash.

The following mixtures of sodium hypochlorite and Textone were tested, all ratios being based on available chlorine:

TABLE I

No. of Solution	*Total Avail. Cl_2 in Solution*	*Ratio NaClO to Textone*	*Fluidity*
1	3 gpl.	1:1	—
2	3 gpl.	1½:1	5.7
3	3 gpl.	2:1	6.1
4	3 gpl.	3:1	6.2
5	3 gpl.	4:1	7.0
6	1.5 gpl.	1½:1	4.8
7	1.5 gpl.	4:1	5.7
Regular chemic	—	—	8.1

Results—White was good on all soured samples, but, as with the regular chemic bleach, it was poor on unsoured samples.

The "fluidity" of each of the soured samples was determined because the greater the fluidity the greater the amount of oxycellulose present.

These results show that the greater the relative amount of hypochlorite used, the higher the fluidity and, undoubtedly, the greater the amount of oxycellulose in the sample.

Solution No. 6, having 1.5 gpl. total available chlorine in solution and a hypochlorite-to-Textone ratio of 1½:1, gave the best results.

pH Tests

Light tag cloth was used in these experiments.

The regular process after kiering was as follows:

Wash in cold water; sour with 1 per cent (approx.) H_2SO_4; wash in cold water; bleach with 0.3 per cent NaClO solution, 3 gpl., cold; stack 30 minutes; wash in cold water; anti-chlor with cold SO_2 water; stack 15 minutes; wash well in cold water; dry.

In the laboratory tests, the same procedure was followed, using the following solutions:

TABLE II

No. of Solution	*Total Avail. Cl_2 in Solution*	*Ratio NaClO to Textone*	*pH*
1	3 gpl.	Straight NaClO	10.8
2	1.5 gpl.	1½:1	10.6
3	1.5 gpl.	1½:1	9.5*
4	1.5 gpl.	1½:1	9.1*
5	1.5 gpl.	1½:1	8.7*

*pH reduced to given point by buffering with sodium bicarbonate.

Results—Sample No. 1 showed considerably more oxycellulose than the rest.

Samples 4 and 5 bleached faster and to a somewhat better color than the rest.

To determine the fiber strength of the samples, the following breaking tests were made, each figure being the average of three breaks.

TABLE III

No. of Sample	*Warp*	*Filling*
Bleached in plant	43 1/3	28
1	43 1/3	28 1/3
2	45 1/3	29 2/3
3	44 1/3	31
4	45 2/3	31 1/3
5	45 2/3	31 1/3

Samples 4 and 5 are definitely stronger than those bleached with straight hypochlorite, i.e.: the sample bleached in the plant and No. 1.

Relation Between Speed of Bleaching and pH—In this work, it was found that the speed of the bleach depends largely on the pH of the bleaching solution, being considerably faster at a low pH (8.7) than at a relatively high pH (9.7 to 10.0). With cloth that has been sufficiently "bottomed," the following values of pH were found most effective for bleaches requiring the times stated:

TABLE IV

Time Required to Bleach	*Best pH*
30 minutes	8.7 to 9.0
2 hours	9.2 to 9.4
6 to 12 hours	9.7 to 10.0

The reason for using a higher pH with slower bleaches is that the available chlorine in the cloth is used up too rapidly at the lower pH to be effective.

Buffering—A mixture of sodium bicarbonate and sodium carbonate was found to work very well as a buffer to hold the pH of the solution constant at the desired point. The following ratios were used:

TABLE V

Parts Bicarb.	*Parts Carb.*	*Required pH*
3	1	8.7 to 9.0
2	1	9.2 to 9.4
1	2	9.7 to 9.9

Conclusions from Tests

1. Acid souring following the kier boil and preceding bleaching improves the color.
2. Only from one-half to two-thirds of the total available chlorine used in the straight hypochlorite bleaching solution is needed when mixture of hypochlorite and Textone is used.
3. To get the best fluidity (showing lowest amount of oxycellulose) the ratio of hypochlorite to Textone should be 1½:1.
4. The best pH for this process is between 8.7 and 10.0: the longer the time required for bleaching, the higher the pH should be.
5. Equipment used for straight hypochlorite bleaching can also be used for hypochlorite-Textone bleaching.

PLANT TRIALS

The results secured in the laboratory with hypochlorite-activated Textone were applied to full-scale bleaching operations in several different plants.

In all these trials, the regular procedure for bleaching with straight hypochlorite was followed when Textone-hypochlorite was employed, and the same equipment was used. The only additional equipment needed was a supply tank for holding the stock Textone-hypochlorite solution and a make-up tank for the buffer solution.

The ratio of NaClO to $NaClO_2$ was in all cases 1½ to 1, in terms of available chlorine.

The only control required is the adjustment of the pH of the solution to the desired point by means of the buffer solution. The pH of the solution can be checked with sufficient accuracy by dipping pH papers into samples of the buffered bleaching solution. The action of the bleach will, however, destroy the pH indicator color almost immediately, and, if it is desired to prevent this, the bleaching action can be killed with hydroquinone before testing. The use of an electrometer will, of course, give more accurate pH readings.

The available-chlorine concentration of the bleaching solution can be checked by titrating it with sodium thiosulfate after adding potassium iodide and acetic acid to the solution.

Plant Trial No. 1—The cloth employed in these trials was high construction broadcloth shirting, 4 yards/#. Three runs were made using hypochlorite-activated Textone instead of 0.25 per cent (2.5 gpl.) NaClO.

No. of Run	*Yards*	*Concentration*	*Temp.*	*pH*
1	2,000	0.1% (1 gpl.)	Cold—80° F.	8.8*
2	2,000	0.1% (1 gpl.)	Cold—80° F.	8.7*
3	10,000	0.1% (1 gpl.)	Cold—80° F.	8.7*

*About 0.3% sodium bicarbonate was used to secure the desired pH.

Results—Whiteness, equal or better than regular. Oxycellulose lower than regular as measured by silver nitrate test.

Plant Trial No. 2—The cloth used in this trial was 48 square, heavy sheeting, 1,000 yards.

Textone Activated bleach solution used, instead of 0.5 to 0.6 per cent NaClO:

Total available chlorine, 0.3 per cent, 3 gpl.
pH, 8.7
Buffer, 0.3 per cent sodium bicarbonate
Temperature, cold, 80° F.

Results—Whiteness slightly better than regular.

Breaks—average of 14 chemic-bleached and 22 Textone Activated-bleached samples (Kier 1):

	Warp	*Filling*
Regular	75.5	63.0
Textone Activated	77.6	64.2

Breaks—average of five samples (Kier 2):

	Warp	*Filling*
Regular	72.1	61.2
Textone Activated	76.3	66.5

Plant Trial No. 3—The cloth used here was 46½", 1.44 yds./# duck, 2,000 yards.

Textone Activated bleach solution used instead of 0.5 per cent (5 gpl.) NaClO:

Total available chlorine, 0.3 per cent, 3 gpl.
pH, 8.8
Buffer, 0.25 per cent sodium bicarbonate
Temperature, cold, 80° F.

Results—Whiteness, very superior to regular

Breaks—two sets of samples.

	Warp	*Filling*
Regular	95	91
Textone Activated	132	95
Regular	110	102
Textone Activated	145	91

Plant Trial No. 4—4 yd./# cloth and pillow tubing.

Textone Activated bleach solution used, 1.0 gpl. av. cl_2.
pH, 9.2
Stacking time—one hour

Results—Excellent in all respects—fluidity average 4.5 to 5.0.

Break Tests on Tubing:

Kier	*Process*	*Fluidity*	*Warp*	*Filling*	
1	Regular	8.40	62.2	56.2	Cloth of similar construction
2	Regular	7.53	58.4	57.4	
3	Activation	4.43	69.8	56.4	
4	Activation	4.78	65.4	57.2	
5	Activation	4.46	70.0	58.4	
6	Activation	4.39	67.6	60.6	
7	Regular	7.44	63.4	72.6	Cloth of similar construction
8	Regular	8.21	61.2	50.2	
9	Regular	8.34	67.8	75.2	
10	Regular	8.20	67.2	66.0	
11	Activation	4.36	75.6	83.4	

Plant Trial No. 5—Heavy sheeting, 1.6 yds./#, 72" wide.

Textone Activated bleach solution used, 2.0 gpl. av. Cl_2.
pH, 9.7.
Stacking time—5 to 12 hours

Results—Excellent in all respects—fluidity average 4.5 to 5.0.

Plant Trial No. 6—Medium weight goods, 4 yds./# approx.

Bleached between boils, and after second boil.

Textone Activated bleach solution used instead of a calcium hypochlorite bleach, 1.5 gpl. av. cl_2.
pH, 8.7-9.0
Stacking time—25 minutes
} both bleaching operations

Results—Very superior to the regular process in regards strength, permanency of white, oxycellulose formation and evenness of shade.

Plant Trial No. 7—Medium and heavy weight bedspreads.

Textone Activated bleach solution used, 1.5 gpl. av. cl_2.
pH, 9.2-9.3
Stacking time—1 hour

Results—Superior to regular process.

REPORT, LOWELL TEXTILE INSTITUTE STUDENT CHAPTER

ON May 21 Ernest P. James was elected Chairman, and Stanley Szopa, Secretary, for the year 1941-42. The Chairman appointed a program committee consisting of Robert McCartney, Chairman, Donald Thomas and Arthur Teichner.

Respectfully submitted,
STANLEY SZOPA, *Secretary.*

ONE HUNDRED AND THIRTY-THIRD COUNCIL MEETING

THE Council held its 133rd meeting at the Chemists' Club in New York on Saturday morning, June 14, 1941. Present were President Carl Z. Draves, presiding; Thomas R. Smith, Vice-President; William R. Moorhouse, Treasurer; Louis A. Olney, Chairman of the Research Committee; William H. Cady and P. J. Wood, Past Presidents; Clarence L. Nutting representing Northern New England; Peter J. Ariente representing Rhode Island; George L. Baxter and Harold W. Stiegler representing New York; William Ebersold and Charles A. Seibert representing Philadelphia; A. Henry Gaede and Ralph E. Rupp representing Piedmont; Robert W. Philip representing Southeast; Bertil A. Ryberg, Associate Director; and Harold C. Chapin, Secretary.

The Secretary's report of the 132nd Council meeting and balance sheet of June 7, and the Treasurer's report of June 11, were accepted. A report from the Treasurer of the Philadelphia Section was approved. A report was received from the Finance Committee, and it was voted that $7,400 be invested in the type of Federal Treasury bond paying interest through maturity at $10,000. Mr. Ryberg's salary was voted, and subsequently he appeared before the Council to report on his activities.

Views of Section Secretaries regarding dues of members in government service were reported, and in accordance with suggestions therein, it was voted that there be published an invitation to any member of the Association to retain his listing as an Association member through such part of the current and next dues year as he might be in the armed forces of the United States, without obligation other than the reporting of such service to the Secretary of the Association; that during such period he be entitled to subscribe to all publications mailed generally to members, upon payment of $2.50 per annum in lieu of regular dues; and that no lapse in payment of dues concurrent with his service be recorded as indebtedness to the Association.

After consideration of letters from the Philadelphia Quartermaster Depot to Hugh Christison and the President, it was voted that a committee be appointed by the Chairman of the Research Committee to confer with a representative of the Quartermaster Depot on specifications for olive drab dyeing of wool.

The Bibb Manufacturing Co., Broad Brook Co., Graniteville Co. and National Carbon Co. were elected to Corporate membership. Each of the following was elected to the class of membership specified, as of thirty days from publication of application, provided that no objection be received by the Secretary:

Senior

R. Abrahams	J. J. Errera	J. S. Roberts
H. C. Allen	B. F. Funderburke	J. J. Rohr
C. E. Beck	J. T. Gillespie	J. S. Sargent
B. S. Bellemere	C. F. Gromann	L. O. Seekatz
A. Bonanno	W. Hofmann	F. W. Senna
W. Boyd	J. J. Hallman	R. E. Sheehan
J. W. Breckenridge	H. F. Logan	F. L. H. Sjostrom
J. J. Cohen	R. T. McDowell	G. A. Stevens
W. D. Corrigan	R. L. McEwen	H. A. Sykes
W. J. Crocker	W. J. Nolan	W. R. Turner
A. D. Damon	S. A. Pettus	J. A. Verhage
H. M. Darling	J. Quance	R. Welsh
J. N. Dow	D. A. Radovsky	I. A. Wood
S. Q. Duntley	R. G. Riley	

Junior

J. A. Fanning	J. R. Gillis	J. R. Luick
G. J. Favro	E. D. Griffin, Jr.	J. N. Murphy
H. A. Gatterman	H. D. Kinsey	J. M. Smith

Associate

W. L. Abramowitz	J. G. Havey	H. E. G. Tanguay
B. D. Anderson	M. L. Hirsch	W. W. Thompson
C. J. Conner	J. R. Simpson	

SECRETARY'S BALANCE SHEET—JUNE 7, 1941

	Applications	*Dues, Regular and Reinstatement*	*Dues, Corporate and Sustaining*	*Miscellaneous*	*Bank Charges*	*Totals*
Brought forward, and received by Secretary, Aug. 1, 1940, to April 18, 1941	1,565.00	$11,587.50	$2,395.50	$3,250.34	—$3.87	$18,793.97
Received by Secretary, April 18 to June 7, 1941	227.50	213.50	550.00	154.15*	— .25	1,144.90
Total received	1,792.50	11,801.00	2,945.00	3,404.49	— 4.12	19,938.87
Transmitted to Treasurer	1,675.00	11,622.50	2,895.50	3,285.24		19,477.74
Leaving to order of Secretary	117.50	178.50	50.00	119.25	— 4.12	461.13

Miscellaneous Items*

Year Books	$55.20	Crock cloth	9.00
Publicity pamphlets	64.25	Moth cloth	10.80
Reprints	4.50	Button	1.50
Reporters	2.85		
Dyed standards	6.05		$154.15

Student

V. A. Baker

The following were transferred from other classifications to Senior membership: R. G. Buck, H. F. Clapham, W. A. Levins, J. R. Redmond, and P. A. Warner.

Respectfully submitted,

HAROLD C. CHAPIN, *Secretary.*

MEETING, RHODE ISLAND SECTION

ON May 23, 1941, Ben Verity was elected Chairman of the Rhode Island Section; Howard V. King, Vice-Chairman; Iver W. Fallstrom, Secretary; Robert R. Farwell, Treasurer; and Edwin Farnsworth, Wilfred A. Lord, Raymond A. Pingree and Harold B. Sturtevant members of the Sectional Committee; all to take office at the Annual Outing at the Norwich Inn, Norwich, Conn., on June 20, 1941. Allison R. Fletcher and Frederic A. Prisley were elected Councilors for the year 1942. Thomas H. Roberts was appointed Chairman of the committee to prepare the Section's paper for the Intersectional Contest at the Annual Meeting of the Association.

Respectfully submitted,

LOUIS D. FLEMING, *Secretary.*

MEETING, SOUTH CENTRAL SECTION

AT a meeting of the South Central Section at the Farragut Hotel in Knoxville, Tenn., on Saturday evening, May 1, 1941, Henry E. Millson of the Calco Chemical Div., American Cyanamid Co., presented his paper on, "Microscopic Observations of the Dyeing of Wool" and George Dempster, Knoxville newspaper columnist and engineer, spoke on the Panama Canal and Canal Zone. Walter E. Hadley was appointed Chairman of the committee for the Annual Outing at Lookout Mt. Hotel on Aug. 1 and 2, 1941.

Respectfully submitted,

FRANK F. MYERS, *Secretary.*

MEMBERSHIP APPLICATIONS

Corporate

National Carbon Co.—Cleveland, Ohio.

Senior

A. Durfee Damon—Chief Chemist, Dutchess Bleachery, Inc., Wappingers Falls, N. Y. Sponsors: F. Kiviat, I. W. Fallstrom.

William Hofman—Supt. of Dyeing, Walther Mfg. Co., Philadelphia, Pa. Sponsors: J. H. Culver, R. F. Culver.

Russell T. McDowell—Salesman, Woonsocket Color & Chemical Co., Woonsocket, R. I. Sponsor: J. H. O'Donnell.

Samuel Atwell Pettus—Salesman, E. I. du Pont de Nemours & Co., Inc., Charlotte, N. C. Sponsors: J. D. Sandridge, J. T. McGregor.

David A. Radovsky—Asst. to Colorist-Chemist, Normandy Print Works, Fall River, Mass. Sponsors: L. B. Coombs, G. L. Joblon.

F. L. Harold Sjostrom—Treasurer and Manager, C. G. V. Sjostrom Co., Springfield, Mass. Sponsors: E. S. Chapin, A. H. Sampson.

Junior

Edmund D. Griffin, Jr.—Chemist, Aridye Corp., Rock Hill, S. C. Sponsors: W. B. DePass, C. S. Powell.

Morris L. Hirsch—Manager, South Pittsburg Plant, Star Woolen Co., So. Pittsburg, Tenn.

Applications for transfer to Senior membership: R. G. Buck, J. R. Redmond.

REPORT, PHILADELPHIA TEXTILE SCHOOL STUDENT CHAPTER

AT the 29th meeting of the Philadelphia Textile School Student Chapter on May 6, 1941, Dr. Donald H. Powers, research chemist of Rohm & Haas Company of Philadelphia, delivered an interesting talk on "Recent Developments in Synthetic Resins and the Applications of Resins to Textiles." The lecture was illustrated by samples of resins, plastics, and resin-treated fabrics. Dr. Powers answered questions raised by the members.

At the 30th meeting held on May 13, 1941, the following men were elected to office for the 1941-42 school year:

Chairman—Joseph E. Dunson.
Secretary—Robert J. Wiley.
Treasurer—Richard A. Ewing.
Sectional Committee:
3rd year—Gordon S. Cohen.
2nd year—Stanley M. Suchecki.

Respectfully submitted,

JOSEPH E. DUNSON, *Secretary.*

ANNUAL MEETING
AND CONVENTION
Carolina Hotel, Pinehurst, N. C.
October 31st and November 1st, 1941

Photochemical Reactions in Silk

(Continued from page 346)

was found that none of the silk samples kept in the dark exhibited any deterioration, showing that the deterioration of the irradiated silk was not thermal, but photochemical.

2. The Effect of Pretreatment with Different Cationic Substances

During processing, such as dyeing, silk is frequently treated with acidic solutions which, as seen from the foregoing experiments, reduces the cation content and increases the susceptibility of the fiber to damage by light. The question whether the stability of acid-treated silk can be increased by replacement of cations thus becomes of importance.

Preliminary experiments showed that basic inorganic substances such as sodium hydroxide or sodium carbonate are effective for increasing the stability of the silk but they impart a harsh, undesirable feel to the cloth. It was observed, however, that basic organic compounds do not produce such undesirable effects and accordingly, a few of these types of compounds were studied. The effect of pretreatment of samples of degummed and electrodialyzed or acid-treated silk with 0.01 *M* solutions of several cationic compounds is shown in Table 2. The data show that the deleterious effect, on light exposure, produced by acidic treatments can be reversed by treatments of the silk with suitable reagents. It is seen that the stability of the silk to light increases with increase in pH of the solutions used for treatment of the silk. This is to be expected, since as shown by the titration curve of silk[6], the binding of cations also increases with increasing pH. In addition to pH, the chemical nature of the cationic substance may also influence the rate of the photochemical decomposition, but sufficient data are not available to enlarge upon this point.

TABLE 2
Effect of Different Cationic Substances on the Deterioriation of Silk

Sample	*Treatment**	*pH of Solution*	*Loss in Breaking Strength on Exposure to Light*
			%
Degummed	.....	..	32
Acid-treated	.....	..	41
Electrodialyzed	.....	..	41
Degummed	dodecyltrimethylammonium bromide (Tech.)	6.0	32
Acid-treated	dodecyltrimethylammonium bromide (Tech.)	6.0	33
Degummed	dodecyltrimethylammonium bromide (Tech.)	9.8	24
Acid-treated	dodecyltrimethylammonium bromide (Tech.)	9.8	26
Degummed	triethanolamine	10.4	26
Acid-treated	triethanolamine	10.4	23
Electrodialyzed	triethanolamine	10.4	26
Degummed	trimethylbenzylammonium hydroxide	11.5	21
Acid-treated	trimethylbenzylammonium hydroxide	11.5	19
Electrodialyzed	trimethylbenzylammonium hydroxide	11.5	19
Degummed	dimethyldibenzylammonium hydroxide	11.5	21
Acid-treated	dimethyldibenzylammonium hydroxide	11.5	21
Electrodialyzed	dimethyldibenzylammonium hydroxide	11.5	21

*The concentration of the solutions was 0.01 *M*. Treatment with more concentrated solutions gave no additional protection.

In general, the inhibiting action of a given compound was independent of the history of a given sample; thus, degummed, acid-treated, or electrodialyzed silks all exhibited the same stability after treatment with a given reagent.

It was noted that the use of a cationic substance in the form of the salt, such as dodecyltrimethylammonium bromide (Tech.) had no effect on the degummed silk although it did raise the stability of the acid-treated sample to that of the degummed sample. Addition of enough dilute sodium hydroxide to raise the pH of the salt solution to 9.8 produced a significant increase in the stability of both, although an equivalent amount of sodium hydroxide alone had no measurable effect on the silk. The reason for this is found in the titration studies of silk[6] in which it was shown that, at a given pH, silk combined with considerably more base in the presence of salts than in their absence.

When the samples, which had been treated with the reagents listed in Table 2, were washed thoroughly with distilled water most of the inhibiting effects of the reagents were eliminated. The stabilities were lowered to about that of the original degummed silk, but they were still higher than those of the electrodialyzed or acid-treated samples. The pH of the water used for washing the samples was about 6.5 and accordingly, the silk would have still retained appreciable amounts of cations[6], which would account for these differences. It may be that Bonicatti's[4] failure to observe the stabilizing effect of cations resulted from his use of degummed silk which might have contained appreciable amounts of cation. This appears likely since he found a marked decrease in the stability of his silk when he pretreated it with acid.

3. The Effect of Methylation

It is shown in the previous sections that the removal of cationic substances from silk by two independent methods results in practically identical decreases in stability to light, but that the stability can be restored by treatment with basic compounds. The question therefore arises whether the decreased stability obtained after electrodialysis or acid treatment is caused by the removal of cations from the carboxylic acid or the tyrosine hydroxyl groups, or from both.

It is not possible to ascertain by a simple experiment whether the cations are combined with carboxyl groups, tyrosine hydroxyl groups, or both. However, it is possible to inactivate known amounts of either type of group with

diazomethane[7]. The results of such a procedure, applied to silk containing 0.002 milliequivalent, or less, of cation per gram, are shown in Table 3. It was found that methyla-

TABLE 3
The Effect of Selectively Methylating Carboxyl and Tyrosine Hydroxyl Group with Diazomethane on the Deterioration of Silk

Sample	*Carboxyl*	*Tyrosine Hydroxyl*	*Loss in Breaking Strength on Exposure to Light*
	% of Total	*% of Total*	%
Electrodialyzed	0.0	0.0	41
I	0	5	35
II	15	16	31
III	31	15	31
IV	67	17	32
V	100	87	23

tion of 5 per cent of the hydroxyl groups produces a significant increase in the stability of the silk. Samples II, III, and IV were approximately equal with regard to the methylation of the hydroxyl groups and equal with regard to their stabilities to light, although they differed greatly with respect to the extent of methylation of their carboxyl groups. This shows that the methylation of the latter has no effect on the stability of the silk. However, a further increase in the methylation of the hydroxyl groups, from 17 to 87 per cent of the total, produced still greater stability. The stabilities attained are comparable to the stabilities of those samples which were treated in alkaline solutions at pH 10.4 or above (Table 2). Some additional evidence suggesting a confirmation of this explanation for an increase in the stability of silk on treatment in basic solutions was obtained in the following way. Portions of samples IV and V were treated with triethanolamine and trimethylbenzylammonium hydroxide and the treated samples were then irradiated for 20 hours. As shown in Table 4, treatment of sample V had little additional effect

TABLE 4
Effect of Cationic Substances on the Deterioration of Samples of Silk Methylated to Different Extents

Sample	*Treatment*	*Loss in Breaking Strength on Exposure to Light*
		%
IV	None	32
IV	triethanolamine, pH 10.4	27
IV	trimethylbenzylammonium hydroxide, pH 11.5	18
V	None	22
V	triethanolamine, pH 10.4	25
V	trimethylbenzylammonium hydroxide, pH 11.5	22

on stability. This was to be expected since most of the hydroxyl groups had already been methylated. On the other hand, the stability of IV after similar treatments was appreciably increased. In this sample, however, a large number of hydroxyl groups were still available for combination with the cations.

The increase in stability of silk, obtained by replacing the hydrogen of the hydroxyl group of tyrosine by methyl groups or by various cations, may result from a change in the absorption spectrum of the silk. The absorption spectra of some simple aromatic hydroxy-aldehydes and hydroxy-ketones are changed by similar treatments[11].

4. Effect of the Irradiation on the Tyrosine Content of Silk

The deterioration of silk fibers by photochemical reactions probably results from a photochemical fragmentation of the polypeptide chains of the protein[3]. Some of the tyrosine may be destroyed[12], [13] and peptide linkages adjacent to the tyrosine cleaved[14]. Destruction of some of the tyrosine was also noted in the present investigation as shown by the data in Table 5. It is of interest to note that

TABLE 5
Photochemical Decomposition of Treated and Untreated Silk Measured by Change in Tyrosine Content and Breaking Strength

Sample	*Tyrosine*	*Loss in breaking strength on exposure to light*
	%	%
Degummed (unexposed)	12.1	..
Degummed + triethanolamine (pH 10.4)	12.0	33
Degummed + sodium hydroxide (pH 10.7)	11.5	53
Degummed	11.1	62
Acid-treated	10.9	77

the decrease in tyrosine content was accompanied by a corresponding decrease in tensile strength, and that treatments which tended to increase the stability of the silk decreased the amount of tyrosine destroyed.

5. Practical Significance

The above observations show that the stability of silk to light can be considerably increased by treatment with suitable cationic reagents. Experiments with several organic bases show that the undesirable effects produced by inorganic bases can be avoided by the use of proper organic reagents. In addition, the latter type of compounds appear to be more efficient in regard to stabilizing the silk. It should be noted that with the exception of the diazomethane treatment, none of the effects of the treatments used in this work was of a permanent nature, since the stabilizing effect was removed by washing the fabrics with water. It may be possible to develop colorless, basic compounds which might have sufficiently high affinities for the fiber so that they would resist removal by washing in natural or alkaline solutions. Whether any of these treatments will affect other properties of the fabric such as light-fastness of the dye which may be used can only be determined for the materials to which such reagents may be applied.

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OBITUARY

ROY L. JONES

ROY L. JONES, director of the sales order department of the Calco Chemical Division, American Cyanamid Co., Bound Brook, N. J., died at his home in Millington, N. J., on June 12th, after an illness of a few weeks.

R. L. Jones

Mr. Jones was born in Charleston, W. Va., and graduated from Harvard University in 1914. Shortly after graduation he went with the Aluminum Ore Co. of East St. Louis, Ill. as a chemist and in 1918 went with the General Electric Co. at Pittsfield, Mass., where he was employed in the electrical research laboratories. At the time of his death he had been with the Calco organization for 21 years.

He was a Mason and affiliated with the East St. Louis Lodge. He was formerly a member of the American Association of Textile Chemists and Colorists.

He is survived by his widow, three children, Christine, Shirley and David, his mother, a sister and a brother.

• TEXTILE FIELD DAY

The Textile Field Day and Golf Outing for the textile, knit goods, and allied trades, will be held at the Westchester Country Club, Rye, N. Y. on Thursday, July 17. Last year, the attendance was over 450 with about 300 participating in golf, and the rest partaking of the tennis matches, and other athletic events. Many of the out-of-town buyers, mill executives and branch-office representatives of New York selling houses have been timing their market visits, for the past several years, so as to be here for the big Textile Outing.

C. Ted Haase of Snider, Bumstead & Company is Outing Chairman, with Lewis M. Davoll of A. S. Haight & Company as co-chairman, and A. L Cassen of Cassen & Bertram as prize committee chairman. Harry Roth is chairman for the knit goods committee; L. Roy Bagge and J. Frank Treene are co-chairmen of the golf committee; Julius Plaut is chairman of the Athletic games committee including soft ball; A. L. Helwith is chairman of the tennis committee.

Harold J. Timmerman of C. P. Lord Company and William H. Bullwinkel of Mooresville Cotton Mills, both of 40 Worth Street, are co-chairmen for the Ticket and Welcoming Committee.

• DCAT SKYTOP MEETING

More than 25 members of the Drug, Chemical and Allied Trades Section of the New York Board of Trade met on June 12, at the Advertising Club for luncheon to draw up the program for the Section's 6th Annual Fall Meeting and Golf Tournament which will be held on October 23-26, at the Skytop Club, Skytop, Pennsylvania.

John J. Toohy, Chairman of the Section, presided. A program similar to that of last year was decided upon. R. F. Berls, Vice Chairman of the Section and General Chairman for the meeting expressed the hope that all Section members who wish to attend this year would make their reservations as soon as possible since the hotel accommodations are limited.

• DIRECTOR, EXPORT DEPARTMENT

Dr. Theodore M. Switz has been appointed director of the Export Department, succeeding P. W. Meyeringh, Hercules Powder Company announced recently. Dr. Switz, formerly assistant director of the department, fills the vacancy caused by the recent election of Mr. Meyeringh as vice president and member of the executive committee.

He became associated with Hercules Powder Company in the Advertising Department in 1937, where he conducted marketing research studies of chemical products. In March, 1938, Dr. Switz was transferred to the Export Department to further the sales of Hercules chemical materials for industry in all parts of the world.

• GENERAL RELEASES

General Dyestuff Corp., 435 Hudson Street, New York City, has released circulars describing the following products:

Acid Anthracene Brown WSGA — a chrome color which is said to combine good level dyeing properties with very good fastness to light, water, sea water, and perspiration. The fastness to washing, fulling and carbonizing is said to be good. It is suitable for dyeing yarns, loose wool and slubbing but is primarily used on piece goods. It is applied largely to the production of fast browns on materials (for example, men's wear) in which silk effects are to be left white. Circular G-265.

Celliton Fast Black BA—a dispersed, direct dyeing acetate dyestuff said to possess good dyeing and penetrating properties. It dyes full shades of bluish black said to possess very good to excellent fastness to light. It also serves as a base for the production of greys. It is stated that black shades show a very desirable retention of their daylight shades under artificial light. Grey shades turn somewhat greener. Circular G-266.

Igepon T Gel—an auxiliary which, as far as chemical nature and properties are concerned, corresponds to Igepon T powder and paste except that it possesses half the effective strength. It is a clear amber-colored thin jelly. It is said to dissolve completely in hot water. Properties claimed for the product are as follows: powerful detergent, stable to hard water, acids and alkalies under conditions met in the textile industry, stable in the presence of salts of most heavy metals, unaffected by bleaching powder, sodium hypochlorite or hydrogen peroxide solutions, preventive agent against lime soap formation and dispersing agent for lime soap deposits, dyeing assistant, wetting out agent which lathers strongly. Circular G-270.

Rapidogen Red FFG—produces bright shades of red said to be of good fastness to light and very good fastness to washing and chlorine. It, like other "FF" brands, is said to show a high speed of coupling, being completely developed by a short ageing. It is suitable for printing on a lactic acid prepare or for printing with Developer RNA, methods which require merely a neutral or vat ageing to completely develop the shade. Circular G-272.

Alphanol Brown BUA — a dyestuff, which, when applied from a weakly acid bath, dyes wool deep shades of brown said to possess fairly good fastness to light and perspiration and good fastness to water and washing. It is used for dyeing fast to washing shades on knitting yarns and loose wool. It is also well suited for dyeing shoddy. It is of further interest for the dyeing of loose fur for the manufacture of hats. Circular G-273.

Rapidogen Scarlet FFR — produces bright, yellowish shades of red said to be of very good fastness to light, washing and chlorine. Like other "FF" types, it is said to show a high speed of coupling, being completely developed by a short acid ageing. It is suitable for printing on a lactic acid prepare or for printing with Developer RNA. Circular G-274.

Rapidogen Orange FFR — produces bright, reddish shades of orange said to be of good fastness to washing and chlorine and very good fastness to light. Other properties are similar to Rapidogen Scarlet FFR, above. Circular G-275.

Rapidogen Red FFBB—produces bright, bluish shades of red said to possess very good fastness to washing and chlorine. Other properties are similar to those mentioned above for Rapidogen Scarlet FFR Circular G-276.

Benzoform Dark Brown VB Extra—a direct color recommended by the manufacturers for the dyeing of cotton and, in particular, rayon. When aftertreated with formaldehyde and acetic acid in the usual way for Benzoforms, it produces deep shades of brown said to be of comparatively good fastness to washing, water and perspiration. It is stated that it is well suited for use on materials which require a degree of fastness to wet processing beyond that obtainable with the average direct color. It is dischargeable to a good white with Rongalite C in neutral pastes but only moderately dischargeable with alkaline pastes. In union dyeing at the boil, wool is dyed somewhat redder and heavier than the cotton, while at about 150 the wool is dyed much lighter than the cotton. Silk effects are dyed; acetate rayon effects are heavily stained. Addition of chrome to the formaldehyde bath turns the shade considerably greener, but does not affect the fastness. Copper sulfate turns the shade slightly yellower and improves the fastness to light. Circular G-278.

Rapidogen Yellow FFGG — produces bright, greenish shades of yellow said to possess good to very good fastness to light and chlorine and very good fastness to washing. Like the other "FF" brands it shows a high speed of coupling, being completely developed by a short acid ageing. It is not suitable for printing on a lactic acid prepare or for printing with Developer RNA. It is of value for producing bright shades of green in combination with Algosol Green IBW Paste Circular G-279.

Celliton Violet 3RA—a dispersed, direct dyeing acetate violet of bright reddish shade. It is said to possess very good dyeing and penetrating properties and to have good affinity, even at low temperatures. It is well suited for dyeing on the jig or in the box. It can be used for dyeing mixed fabrics in which cotton or viscose is to be left clean, since the stain it produces on these fibers during the dyeing is easily removed by an aftertreatment with Blankit I or by a mild bleach with potassium permanganate and sodium bisulfite. Circular G-280.

● DIRECTOR OF RECREATION

Colonel J. Ebert Butterworth, vice-president and treasurer of H. W. Butterworth & Sons Co., entered U. S. Army Service on June 24th as director of recreation at Camp Ritchie, Maryland. He has been granted a one-year leave of absence from the Butterworth company.

©Bachrach

Col. J. E. Butterworth

Colonel Butterworth has been treasurer of the Butterworth organization since 1919. With J. Hill Zahn, he opened the Butterworth southern office in 1923 and represented the company in the South from 1923-30. Since 1920, he has been in the home office in Philadelphia.

Colonel Butterworth was named Captain of Infantry on August 8, 1917, on completion of his course at the First Officers Training Camp at Fort Niagara, N. Y. He was assigned to the command of Company F, 313th Infantry at Camp Meade, Md., and in July 1918, took his Company to France. He served with both the 316th and 313th Infantry Regiments, commanding first a company and then a battalion in action. He was wounded in action at Montfaucon.

He was discharged at Camp Dix in June 1919. In July 1920, he accepted a commission as Captain of Infantry in the U. S. Army Reserves. In 1922 he was assigned to the 316th Infantry with rank of Major. Later in the same year, he was promoted to the grade of Lt. Colonel. On June 14, 1932, he was promoted to the grade of Colonel and was assigned to command of 1301st Service Unit.

Colonel Butterworth graduated from Princeton in the class of 1916.

● AMMONIUM THIOCYANATE

Large scale production of ammonium thiocyanate is now under way at the Everett, Mass., plant of the Eastern Gas and Fuel Associates, it is announced by Fred Denig, vice president of Koppers Comapny in charge of the Research Department.

The plant has large annual capacity and is built to produce both crystals and 30 per cent liquor. The grade of crystals now under production is said to have a purity in excess of 95 per cent.

The chemical is being produced in connection with coke plant operations, from constituents of manufactured gas and this fact is not only said to assure a dependable supply on a scale not heretofore available in this country, but also to make it possible to lower decidedly the price of this chemical. This new United States production, it is stated, will make American industries independent of foreign sources of supply.

The production of the plant will be marketed by the Tar and Chemical division of Koppers Company, Koppers Building, Pittsburgh, Pa.

The versatility of ammonium thiocyanate is indicated by the fact that while it is generally employed by the chemical industry as a reagent and as a starting point for a number of other chemical compounds, it is also used in resin manufacture; in the textile industry for the dyeing, finishing and printing of cloth; in the metallurgical industry for the finishing and pickling of metals; in photography; in pharmacy; as a chemical for insecticide manufacture; and as a weed killer.

● APPOINTED CHEMICAL DIRECTOR

Dr. Joseph D. Clark has been appointed Chemical Director of the Glyco Products Co., Inc., Brooklyn, N. Y., where he will be in charge of research and development.

Dr. Clark first served as a textile consultant and with the U. S. Army Engineers, and later with the Chemical Warfare Service in World War I. Later he was chemical inspector for the State of South Carolina. Then he taught at Syracuse University for seven years before engaging in industrial research and development with the Firestone Tire & Rubber Co., Kalamazoo Vegetable Parchment Co., Flood & Conklin Mfg. Co., and the Nixon Nitration Works.

Dr. Clark is especially versed in applications of the newer chemicals, resins and waxes to the paper, rubber and coating industries. He has spent the last three years working out special application problems, not only in the laboratory, but at the plants of many manufacturers in the East and Middle West.

● BERNARD COLOR CARD

Bernard Color & Chemical Corporation announces the publication of a new color card and dyeing guide dealing with Bernacyl and Bernacyl Diazo colors for acetate-rayon. This card incorporates a variety of suggestions and requests that practical dyers have made.

All colors have been illustrated in two depths and at two temperatures, showing the yield obtained at 160° F. and at 190° F. The descriptive matter also deals with various methods of dyeing and the handling of many types of fabrics.

Fastness tabulations have been so compiled that they correspond to the standards set up by the A.A.T.C.C. Properties on gas fading and sublimation are included in these fastness tabulations.

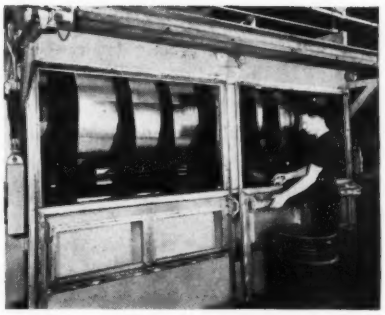
A separate section of the card deals with the dyeing of the Bernacyl Diazo colors and introduces the suggestion of pH control in the developing bath. Also included in this section is a series of practical cautions and remedies to be observed under mill conditions in diazotizing and developing of these colors.

Copies of this guide book have been sent to all textile schools. Copies are available to firms engaged in the dyeing industry upon request.

• NEW PIECE DYE KETTLE DESIGN

Riggs and Lombard of Lowell, Mass., manufacturers of textile finishing machinery, have recently perfected a new Type TLBT Stainless Piece Dye Kettle which they claim is the biggest advance in dye kettle design in 15 years. The reasons for the above claim are as follows:

First, it is stated that the machine has proved to be the nearest thing to a universal kettle that has ever been offered. It has been in the process of development for about 15 months, during which time it has been installed in practically every different type of woolen and worsted mill and is said to have successfully dyed fabrics ranging from 5 oz. flannels to 32 oz. meltons; from worsted shirtings, through gabardines, to heavy men's wear serges; and from knitted shoe linings to heavy knitted ski cloth.



Type TLBT Dye Kettle

Second, it is stated that in no case where these trial machines have been operating have there been any tangles or tie-ups. This is in part due to the peculiar design of the tub which is narrow from front to back, and in part to a large drum type reel with partially knurled surface (patent pending).

Third, for similar reasons, goods balloon, move around and are said to change folds more readily than in any other kettle so that wrinkles, creases, and dye streaks are virtually eliminated.

Fourth, it is claimed that the knurled drum reel absolutely eliminates slip, even on rayon gabardines, and therefore all of the operating troubles which have been caused by slippage. Knurled humps are added to the reel where the type of fabric so requires.

Fifth, the ratio of dye liquor per lb. of cloth in this kettle is so exact that substantial savings in dyestuffs are said to result, even when using acid and chrome colors, while the savings with direct and union colors are proportionately greater.

• TRANSFERRED TO S. C.

Donald S. Collard, Superintendent for a number of years in charge of manufacturing at the Warwick Chemical Company, West Warwick, Rhode Island, has been transferred to their Rock Hill, South Carolina plant with the title of Resident Manager.

•TECHNICAL NOTES FROM FOREIGN SOURCES•

Specifications for a Textile Oil

W. Garner—J. Soc. Dyers and Col., 56-333, *Aug.*, 1940. —The first part of this paper is concerned with the reactions which take place when an ester of an unsaturated fatty acid of the kind occurring in large proportions in many suitable wool combing oils is oxidized by air or oxygen. A typical example of such an ester is ethyl oleate.

Reactions which occur most readily when an ester of this type is oxidized in the vapor phase, i.e., when the molecules are far apart, are not necessarily the same as reactions occurring most readily when the molecules are relatively continuous, e.g., when the oil is in the liquid or solid (chilled) state. The surface/mass-ratio is an important factor in deciding the speed and type of oxidation.

In the vapor phase each molecule may be considered to oxidize as a separate unity, and the oxidation of ethyl oleate, for example, may occur either at the double bond or in the saturated portion of the chain. When the oil is oxidized in the atomized state or as a thin film, reactions between molecules may be expected as an additional feature.

The molecules at the surface of the oil mass will be most readily oxidized and a surface skin of oxidized molecules will form. Individual oxidized molecules will penetrate into the interior of the mass by thermal agitation, being replaced at the surface by unoxidized molecules. The more accessible the oxygen, the more simple are the oxidation products. As the surface/mass-ratio for the oil mass decreases, the likelihood of condensation products being formed increases; these may include dimers, aldehyde-polymers and aldol condensation products.

The author's view is that the oxidation of oils proceeds by a "chain mechanism." According to this view, molecules of esters of unsaturated fatty acids are capable of becoming "activated" and when so activated can react with oxygen, but not otherwise. The first step in oxidation is *chain initiation*, by which is meant the activation of one molecule by addition of externally supplied energy in a mass of inert molecules. The second step is *chain propagation*, which consists of the stages (A) reaction between an activated molecule and oxygen and (B) the activation of an adjacent inert molecule by the liberated energy resulting from (A). The new activated molecule then repeats the cycle. The third step is *chain termination*, which results when an activated molecule takes part in some reaction other than the particular one required to continue the chain.

The tendency towards oxidation shown by fatty esters free from impurities increases in proportion to the unsaturation and in the series stearic, oleic, linoleic, linolenic and clupanodonic acids increases rapidly in proportion to the number of double bonds (0, 1, 2, 3, 4 respectively) in the fatty acid chain. Oils composed mainly of oleic acid esters, e.g., olive oil, have a small tendency to oxidize when free from impurities. It can be shown, however, that an olive oil can be prepared which will oxidize much more

rapidly than a suitably prepared linseed oil, and the rate of oxidation shown by any particular oil cannot be taken alone as any criterion whatever of its suitability as a textile oil. The rate of oxidation gives no indication of the type of oxidation product and depends far more on the amount of certain types of impurities than on chemical constitution.

The more important factors influencing oxidation are now discussed individually. (1) Surface/Mass-Ratio: The speed of oxidation of a mass of oil depends *inter alia* upon the surface exposed to air. In the vapor phase each molecule is exposed to attack by oxygen, and the surface/mass-ratio is, in effect, infinity. When an oil is atomized and oxidized in the form of spray the surface is still very large. When spread on wool as in woolcombing, 1 lb. of oil covers a maximum possible area of about half an acre. It is therefore obvious that such use of oil provides very faborable conditions for oxidation to take place.

(2) Temperature: Oil oxidation, if it follows a well-known chemical rule, about doubles its speed for every 10° rise of temperature. In the Mackey test oxidation curves usually show a sharp rise at 110° C., when the oxidation is about 500 times as rapid as at 20° C. At normal temperatures, oxidation of oil probably always begins by peroxide formation at a double bond. Formation and decomposition of peroxides proceed simultaneously; it is thus only possible to measure the rate at which peroxides accumulate—the P.A.R., or peroxide accumulation rate. Such accumulation is an excellent measure of the *rate* at which an oil oxidizes, but gives no information about the *type* of oxidation products, which is equally important from the textile point of view. The Mackey test examines the tendency of an oil to oxidize at 100° C. There are good reasons for assuming that this test indicates the rate of peroxide formation at second hand by exhibiting the heat produced on decomposition of peroxides, while the P.A.R. test gives direct indication of the process with which all oil oxidation under textile conditions begin the formation of peroxides.

(3) Catalysts: A perfectly pure oleic ester oxidizes comparatively slowly. The rate of oxidation is influenced very greatly by catalysts, and can be increased a million fold in this way. Almost all oils prepared from natural sources contain oxidizing catalysts, of which the most powerful are compounds of the metals occupying the lowest points on the atomic volume curve. Fatty acids are, weight for weight, much less active than metals, such as iron, but have a very considerable effect when present in the percentages commonly found in textile oils. When metallic catalysts and fatty acids are both present their combined effect is very great. Exposure to light has also an important catalytic effect.

(4) Antioxidants: A wide range of compounds, of which ferric oleate is an example, can act as antioxidants. In some cases a body is a catalyst in small amounts and an antioxidant in large amounts. Antioxidants act by breaking an oxidation chain, most probably by their molecules colliding with activated oil molecules and becoming themselves activated, the antioxidant thus in many cases serving merely as a leak mechanism to drain away energy from activated molecules to the oil mass. Artificial antioxidants disclosed in patents are usually phenols, amines or aminophenols. The author has found that compounds that can be considered as derivatives of thioformaldehyde were excellent for this purpose. The rate of oxidation of non-drying oils is entirely determined by the balance between the catalyst and the antioxidant activity.

It is extremely difficult to draw up a specification which will exclude all oils likely to be deleterious. One for combing oils should give information about: (1) undesirable unsaturation; (2) oxidation history; (3) stability; (4) purity; (5) performance. These individual points are discussed in detail, with special attention given to undesirable oxidation products which may be formed, oxidation rate, use of the Mackey and P.A.R. tests in determining stability, and behavior of an oil during combing, drawing and spinning, as well as its scourability and absence of discoloration. Numerous graphs and tables are included in the article.

The Dissolution of Chemically Modified Cotton Cellulose in Alkaline Solutions

Brownsett, Davidson and Clibbens—Jour. Text. Inst., XXXII, 2, T15, Feb., 1941.—Under this general title appear two papers, Parts 6 and 7 in a series from the laboratories of the British Cotton Industry Research Association. Part 6, by T. Brownsett and G. F. Davidson, deals with the effect of the method of modification of cotton cellulose on the relation between its fractional solubility in sodium hydroxide solution and fluidity in cuprammonium solution; Part 7, by T. Brownsett and D. A. Clibbens, considers the solvent action on modified cotton of solutions of trimethylbenzyl—and dimethyldibenzyl ammonium hydroxides (Tritons B and F).

(Part 6) In a previous paper it had been shown that the relation between fractional solubility of chemically modified cotton cellulose and fluidity in cuprammonium solution was not the same for all types of modified cotton, but depended on the method of modification and on whether the cotton had been mercerized before modification. The present paper describes a more comprehensive investigation of the effect of the method of modification on the solubility-fluidity relation, the materials studied comprising nine different types of modified cotton. The solubility measurement employed was the determination of the percentage of the modified cotton dissolved by 2.5 N sodium hydroxide solution at —5° C. Under these conditions an unmodified cotton is almost completely insoluble and the most highly modified materials show a solubility approximately 100 per cent, so that the whole of the solubility scale is utilized.

The modified cottons employed consisted of a series of hydrocelluloses and eight series of oxycelluloses prepared by the action of (1) alkaline hypobromite, (2) alkaline hypochlorite, (3) neutral hypochlorite, (4) acid hypochlorite, (5) dichromate in the presence of sulfuric acid,

(6) dichromate in the presence of oxalic acid, (7) periodic acid and (8) metaperiodate.

Relations determined between alkali solubility and fluidity for the various types of modified cotton are shown graphically, and tables are employed to display the effect of pretreatments with hot and cold alkali solutions on this solubility and fluidity. Experimental procedure is described, and results thus summarized:

Oxycellulose prepared by the action of alkaline hypobromite or alkaline hypochlorite, or of dichromate in the presence of sulfuric or oxalic acid, give approximately the same fluidity-solubility relation as hydrocellulose, but neutral hypochlorite and acid hypochlorite oxycelluloses are considerably more soluble, and periodic acid and metaperiodate oxycelluloses considerably less soluble, than hydrocelluloses of equal fluidity. Treatment of the modified cotton with cold or hot dilute sodium hydroxide solution produces changes of fluidity and solubility that vary in magnitude from one type of modified cotton to another, and after these treatments the relation between the two properties is the same whatever the method of modification. Periodic acid and metaperiodate oxycelluloses are exceptional in showing relatively large increases of solubility as a result of pretreatment with cold dilute sodium hydroxide solution, and this behavior is ascribed to the presence in these oxycelluloses of alkali-sensitive linkages that are not broken by the relatively short alkali treatment involved in the solubility measurement. The abnormal fluidity-solubility relation given by the neutral hypochlorite and acid hypochlorite oxycelluloses is due to an abnormality in the behavior of these types of modified cotton towards cuprammonium.

(Part 7) Previous papers contain measurements and discussion concerned with the partial solubility of chemically modified cellulose in solutions of lithium, sodium, potassium and tetramethylammonium hydroxide, and in cuprammonium and cupri-ethylenediamine solutions, at 15° C. and lower temperatures. Solutions of certain quaternary ammonium hydroxides are better solvents for cellulose than the tetramethyl base or hydroxides of the alkali metals. The use of benzyl-substituted ammonium hydroxides as cellulose solvents was patented recently (U. S. P. 2,009,015/1935), and solutions of trimethylbenzyl- and dimethyldibenzyl-ammonium hydroxides became commercially available under the names of Triton B and Triton F, respectively. The methods used for other bases have now been applied in a study of cellulose solubility in the Tritons.

Several graphs are employed to show the solubilities of both modified and unmodified cottons in solutions of Tritons B and F at different temperatures and concentrations, the relation between fluidity of modified cottons and fractional solubility in different bases at optimum concentrations, and the relative viscosities at 20° C. of aqueous solutions of the different bases examined. Experimental procedure, covering treatment of cotton samples, solvents and methods of determining solubility and viscosity, is described, and results expressed in a series of tables.

Conclusions may be in part summarized: The solubility/concentration curves for Tritons B and F at 20° C., like that for tetramethyl-ammonium hydroxide, contain two maxima. The maximum solubility rises, and the concentration at which it occurs falls, when the temperature is lowered from 20° C. to 0° C. In this respect the Tritons resemble all the other strong bases of which the cellulose solvent powers have been examined. Triton F completely dissolves "unmodified cotton." The first maximum in the solubility curve with Triton F occurs at a base concentration of about 1.9 N, and this is lower than the normal concentration of maximum solvent power for any other base so far examined. A solution of unmodified cotton in Triton F can be diluted until the concentration is as low as 0.5 N without precipitation of the cellulose, though the solubility of cellulose in this concentration of Triton F determined by direct examination is zero.

The Spinning of Dyed and Bleached Yarns

Dr. G. Ullman—Jour. Text. Inst.—XXXII, 2, pl., Feb., 1941.—The spinning of bleached and dyed cotton and mixtures of cotton and staple fiber on machines with three lines of rollers has been practised for some years in many countries and increasingly developed. In this article the author describes the steps of bleaching, dyeing and spinning involved in this rather complicated branch of yarn production.

There is an increasing demand among consumers for variety in fabrics in regard to color, texture and design. To satisfy this demand the methods of production must be the cheapest possible. In many countries printing has had a great vogue, but there are many advantages in colored woven fabrics, and methods of producing them cheaply but in manifold variety are highly important. The use of yarn spun from colored or bleached fiber offers many possibilities in this field, and the present article is intended to give the results of practical experience in the manufacture of such yarns.

In a general consideration of the subject of colored yarn spinning, stress is laid upon the basic fact that spinning is influenced by the surface properties of the fiber. Different types of cotton have different properties; some, like Indian cotton, are rough, others smooth. These effects are displayed in the clinging of the fibers and their behavior in drafting. In spinning colored cotton it is soon realized that a very small percentage of cotton of a particular surface property in a mixture is sufficient to improve or lessen the quality of the spun yarn. In the early days, when extracts or mordant colors, especially basic dyes on tannin-metal mordant, were more widely used, it was common experience that dyed cotton could be spun with 20-30 per cent fewer turns than the same cotton in the gray, because the surface of the dyed fiber was rougher. On the other hand, cotton dyed by a process that involved oiling, as in the case of Turkey red or naphthol colors, or by a process

requiring a strongly alkaline bath, such as Indigo, became so smooth that it was almost impossible to spin. A similar inconvenience is encountered with staple fiber, especially if it has been treated with a softener.

Many years ago the author discovered how to overcome such troubles by blending with the mass a small amount of a smoother or rougher fiber, as required; taking advantage of the fact that such an admixture can be rapidly incorporated with the bulk so as to influence the whole mass. Experience gained in spinning tannin-mordanted dyed sliver, which was too harsh and rough, and Para red dyed fiber, which was too smooth, led to a solution of the problem. Oiled cotton was given in the dye house a final treatment with weak tannin or other liquors to impart a harsh surface, and the harsh tannin-mordanted cotton was given a final oiling treatment. The importance of surface properties of the cotton is demonstrated further by the fact that the author has not been able to spin mercerized cotton. The removal of the cuticle renders the fibers so smooth that no impregnation of the above kind to make a harsh surface was successful.

Under the individual headings of (1) Dyeing and Bleaching, (2) Spinning, (3) Fabrics and (4) Questions and Cost the author deals at length with practical details of colored yarn spinning. Various processes and machines are discussed, and the different sections contain a large amount of technical information.

Quantitative Determination of Indigo on the Fiber

J. Lotichius—J. Soc. Dyers & Col., 56-435, *Oct.*, 1940. —It is very simple to determine quantitatively the Indigo content of dyed cotton by colorimetric Indigo tests.

A piece of the Indigo dyed fabric 5 cm. square is treated in a porcelain dish with 20 cc. of cold concentrated sulfuric acid for from 30 to 45 minutes. The Indigo and cotton both dissolve, forming a dark green solution of Indigo sulfate, which is added to about 750 cc. of cold distilled water in a liter beaker. A good colloidal solution of Indigo is formed, which can be kept for some time. The solution is made up to one liter and its Indigo content colorimetrically determined.

The standard solution for reference may be made by dissolving exactly 10 mg. of chemically pure Indigo in 10 cc. of sulfuric acid (sp. gr. 1.84) and diluting to 1 liter. Instead of this standard solution, which cannot be kept indefinitely, the standard solution of Sirius dyes, used by the author for determining the Indigo content of Indigo vats, may be employed with equally good results.

This colorimetric method is simpler than the test described by Knecht (1909) and can be easily carried out in 1½ hours. It gives very accurate results.

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